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FINAL SUMMARY TECHNICAL REPORT

TO

BUREAU OF NAVAL WEAPONS RRMA-232

NAVY DEPARTMENT

CATALOGED BY SECTION

DESIGN DATA STUDY

FOR

COATED COLUMBIUM ALLOYS

CONTRACT No. N0w 62-0098-c

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TAPCO

A DIVISION OF

*Thompson Ramo Wooldridge Inc.*

CLEVELAND 17, OHIO

DESIGN DATA STUDY FOR  
COATED COLUMBIUM ALLOYS

21 January 1963

Prepared Under Navy, Bureau of Weapons

Contract NOW 62-0098-c

FINAL SUMMARY TECHNICAL REPORT

21 December 1961 through 21 December 1962

Materials Processing Department  
TAPCO a division of  
Thompson Ramo Wooldridge Inc.  
23555 Euclid Avenue  
Cleveland 17, Ohio

ER-5185

FOREWORD

This Final Summary Report is submitted by Materials Processing Department, TAPCO a division of Thompson Ramo Wooldridge Inc., in accordance with the provisions of Contract NOW 62-0098-c. The work was administered under the direction of the Bureau of Naval Weapons, Navy Department, with Mr. Irving Machlin as project engineer.

This report describes the results of the program during the period 21 December 1961 to 21 December 1962.

The program was managed and directed by R.A. Jefferys. J.D. Gadd was in charge of conducting the technical effort and was supported by D.B. Warmuth in the mechanical testing phases of the program.

This report was:

Prepared by

J. D. Gadd  
J.D. Gadd, Metallurgist

Approved by

R. A. Jefferys  
R.A. Jefferys, Research Supervisor

Approved by

A. H. Grobe  
A.H. Grobe, Department Manager

# ABSTRACT

Design data properties were determined for two protective coating-columbium alloy systems involving Pfaudler and TRW oxidation protective coatings on Fansteel-85 columbium alloy up to temperatures of 2600°F. Prior to the design data study, preliminary screening oxidation, bend and prestrain tests were conducted on seven oxidation protective coatings applied to D-14 and FS-85 columbium alloys. Based on the screening test results, Pfaudler and TRW coatings were selected for the mechanical properties evaluation. The following design properties were determined for uncoated and Pfaudler and TRW coated 30 mil FS-85 alloy sheet: (a) coating cyclic oxidation protective life, (b) tensile properties of uncoated (in vacuum) and coated (in air) sheet, (c) coating deformation tolerance and (d) stress rupture properties of coated sheet (in air).

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DESIGN DATA STUDY FOR COATED COLUMBIUM ALLOYS

1. INTRODUCTION

As refractory metal technology becomes more advanced, the utilization of refractory metal components in operational space and re-entry vehicles becomes a more practical problem. Columbium alloys, because of their high temperature strength and low temperature fabricability, show considerable potential as structural materials. The development of surface alloy coatings for protecting columbium alloys from detrimental oxidation and internal contamination in oxidizing environments has been quite successful on a laboratory scale. However, the practical utilization of any coated columbium alloy depends upon the successful union of the coating and base metal to produce a system with useful properties. Until recently, all available data has been generated on independent coating development programs and in the absence of standardized evaluation methods almost no comparison could be made of the properties of various coatings and coating-base metal systems. In order to effectively assess the high temperature capabilities of coated columbium alloys for design requirements, truly comparative mechanical property data for coating-base metal systems must be made available to the design engineer.

A program was initiated by the Bureau of Naval Weapons under Contract NOW 62-0098-c to conduct a comparative evaluation and design data study of presently available protective coatings and columbium base materials. The initial phase of the program involved a series of screening tests. Coatings supplied by various organizations were subjected to comparative oxidation tests and bend ductility tests before and after oxidation, under a variety of test conditions. From this screening analysis, the two most promising coatings were selected in conjunction with the project monitor, Bureau of Weapons, for application to FS-85 columbium alloy for a thorough evaluation of coating-base metal properties. The design data study involved a thorough evaluation of the tensile, stress rupture and strain tolerance properties of the two coating-base metal systems in air at temperatures up to and including 2600°F.





## 2. SUMMARY

A testing program was completed in which comparative mechanical property data was generated for two coating-base metal systems involving FS-85 alloy. The initial phase of the program was comprised of screening tests designed to produce comparative coating property data on all available columbium coating systems. From this preliminary screening evaluation two coating-base metal systems were selected for the comprehensive design data study. Coatings were applied to D-14 alloy by: Chromalloy, McDonnell, Pfaunder, Pratt & Whitney (Canel), GT&E, TRW and Vought and subjected to the following five evaluation tests: (1) metallographic, (2) cyclic oxidation, (3) room temperature bend, (4) cyclic oxidation followed by room temperature bend and (5) room temperature bend prestrain followed by cyclic oxidation.

The coatings ranged in thickness from 2 to 4 mils. Cyclic oxidation protective lives in excess of 150 hours were obtained at 1600°F with all coatings except those applied by McDonnell and GT&E. At 2000°F only the Vought and TRW coatings were protective after 150 hours of cyclic oxidation and only the TRW coating protected D-14 alloy for 150 hours at 2300°F. At 2600°F maximum protective lives of 20-40 hours were obtained with the GT&E and TRW coatings.

Room temperature bend tests on as coated D-14 alloy indicated the Chromalloy, Pratt & Whitney and Vought coating treatments embrittled D-14 alloy for deformation at the normal handling temperature. Room temperature bend tests were also conducted on coated specimens after cyclic oxidation for 50 hours at 2000°F and 10 hours at 2600°F. With the exception of the McDonnell and GT&E coated specimens, all other coatings protected the D-14 alloy substrate against embrittlement in the absence of localized coating failures in the bend section.

Coated D-14 alloy specimens were prestrained at room temperature to bend angles of 2, 5 and 10 degrees and subsequently cyclic oxidation tested to failure at 1600 and 2600°F. Bending to angles of 2 and 5 degrees had no detrimental effect on the protective lives of the seven coatings tested, even though permanent deformation of the substrate was encountered on bending 5 degrees. Room temperature prestraining to a bend angle of 10 degrees produced premature coating failure on all coated specimens at both 1600 and 2600°F.

Based on the performance of the seven coatings in the screening tests over the entire 1600 to 2600°F temperature range, the Vought, Pfaudler and TRW coatings were tentatively selected for the design data study. FS-85 alloy, one of several columbium alloys of current interest to the Materials Advisory Board and to the Bureau of Weapons, was selected as the sheet alloy for the design data study.

Prior to coating the large number of specimens for the design data study, the Vought, Pfaudler and TRW coatings were applied to a limited number of FS-85 alloy specimens for a brief preliminary analysis similar to the previously discussed screening tests on D-14 alloy. No anomalies were observed in the protective characteristics of the three coatings on FS-85 alloy as compared to their performance on D-14 alloy. However, when failure occurred at 2600°F the Vought coating formed a molten corrosive slag which destroyed both the test specimens and support materials. Based on this high temperature limitation of the Vought coating the Pfaudler and TRW coatings were selected as the only coatings to be evaluated in the design data study.

The objective of the design data study was to generate useful and comparative design property data on the two coating-base metal systems and to compare the mechanical properties of the coated substrate in air with those of the uncoated sheet in vacuum. The five areas investigated were as follows: (1) cyclic oxidation tests, (2) thermal shock-erosion-oxidation tests, (3) tensile properties, (4) coating strain tolerance and (5) stress rupture tests.

The TRW coating was protective on FS-85 alloy for in excess of 150 hours of cyclic oxidation exposure at temperatures from 1600 to 2500°F and for a maximum of 51 hours at 2600°F. Protective lives in excess of 150 hours were obtained with the Pfaudler coating at 1600 to 2000°F; however, the coating was protective for only 48-56 hours at 2300°F and 1-6 hours at 2500 and 2600°F in cyclic oxidation.

Thermal shock-erosion-oxidation tests utilizing oxyacetylene torch heating and air blast cooling were conducted on three specimens representing each coating. All specimens cycled from 2600 to 250°F for 100, 250 and 500 cycles failed within 2 hours upon post oxidation at 1600°F.

The tensile properties of uncoated and Pfaudler and TRW coated FS-85 alloy were determined for the temperature range of room temperature to 2600°F. The uncoated sheet was tested in vacuum utilizing tantalum element radiant heating and the coated sheet in



air utilizing quartz lamp radiant heating. Both coatings drastically reduced the FS-85 alloy ductility and ultimate tensile strength at 1200 and 1600°F; however, at temperatures both above and below this range the mechanical properties of the uncoated and coated sheet were nearly comparable. The tensile properties of both coating-base metal systems were nearly comparable over the entire room temperature to 2600°F temperature range.

The coating deformation tolerances of the Pfaudler and TRW coatings were determined by prestraining tensile type specimens in both the elastic and plastic deformation ranges at temperatures from room temperature to 2600°F, followed by a post oxidation treatment for 2 hours at 1600 and 2600°F. Both coating-base metal composites were capable of prestraining into the base metal plastic deformation range at all test temperatures without the loss of 2 hour protection at 1600 and 2600°F. Specimens of both coatings tolerated prestrain in excess of 30% at 2600°F without subsequent post oxidation coating failure.

Stress rupture tests were conducted on Pfaudler and TRW coated FS-85 alloy in air at 1600, 1800, 2000, 2300, 2500 and 2600°F. Four to five stress levels ranging from 20 to 90% of the base metal yield strength were investigated for maximum exposures of 150 hours at each temperature. The Pfaudler coating was protective at several stress levels for in excess of 150 hours at temperatures up to 2000°F and the TRW coating for various stress levels at temperatures up to 2500°F. Stress rupture lives with both coatings under non-thermal cycle conditions at 2600°F and with the Pfaudler coating at other temperatures were in excess of the cyclic oxidation protective lives at these same temperatures. This indicates that subjecting the coated substrate to creep producing stresses during exposure at these temperatures was not as severe as intermittent thermal cycling.

### 3. MATERIALS

Several candidate alloys were considered for the initial coating screening evaluation tests. However, since these tests were only a preliminary to a more thorough evaluation of the more promising protective coating systems, the alloy selection was based primarily upon availability of a representative sheet alloy. Approximately one pound (100 in<sup>2</sup>) of 30 mil D-14 alloy (Cb-5Zr) sheet was purchased from duPont Company. The ready availability of D-14 sheet permitted preparation and shipment of the test specimens to the various coating organizations in a minimum of time.

Table 1 shows the chemical analysis of this heat of material as reported by the vendor.

Fansteel 85 alloy (Cb-28Ta-10W-1Zr) was selected as the sheet alloy for evaluation in the design data study portion of the program and 1827 square inches of 30 mil sheet was purchased from the Fansteel Metallurgical Corporation. Again the time schedule of the program and the availability of sheet material governed the alloy selection from the number of candidate alloys of current interest to the Materials Advisory Board and the Bureau of Weapons. The chemical analysis for this heat of material is also given in Table 1. The high carbon level (250 ppm) in this heat was not detected until after the preliminary evaluation tests had been completed on specimens prepared from this sheet. The original chemical analysis reported by Fansteel for this heat indicated a carbon content of 50 ppm. The results of these screening tests initiated a re-investigation of the heat chemistry by Fansteel. The influence of this high interstitial level on the alloy ductility will be discussed in Section 4.5.

#### 4. PRELIMINARY SCREENING EVALUATION

Several protective coating systems are currently under investigation for the protection of columbium base materials against atmospheric attack during service at elevated temperatures. In order to select from this group the most promising coating systems for a thorough evaluation of coating-base metal mechanical properties, a series of screening tests were conducted to establish directly comparable coating property data. To achieve truly comparative results, specimens representing each of the candidate coatings were group tested at the same time, in the same facilities, and under identical test conditions, in each of the individual tests discussed below. The five relatively simple tests employed in the screening study were as follows:

- (1) Metallography
- (2) Cyclic oxidation tests in air at 1600-2600°F
- (3) Room temperature bend ductility tests on coated and uncoated sheet
- (4) Room temperature bend-prestrain tests followed by cyclic oxidation at 2000 and 2600°F



TABLE 1

Chemical Analysis of D-14 and FS-85 Alloys  
Reported by the Suppliers

<u>Element</u>	<u>D-14(1)</u>	<u>FS-85(2)</u>	
		<u>Ingot</u>	<u>Sheet</u>
O	207 ppm	90 ppm	150 ppm
H	1 ppm	-	-
N	17 ppm	30 ppm	20 ppm
C	29 ppm	20 ppm	250 ppm
Ta	475 ppm	28.21%	27.92%
W	-	11.05%	10.0 %
Zr	4.8%	0.97%	0.98%
Fe	-	<0.005%	-
Ni	-	<0.005%	-
Si	-	<0.005%	-
Ti	-	<0.005%	-
Cb	Balance	Balance	Balance

(1) 30 mil sheet - DuPont - Heat No. 14-114

(2) 30 mil sheet - Fansteel - Heat No. 85D-633

- (5) Oxidation exposure at 2000 and 2600°F followed by room temperature bend ductility tests.

These tests were conducted on seven coatings applied to D-14 alloy specimens. Every effort was made during testing and data processing to conduct a completely unbiased and thorough evaluation of the candidate coating-base metal systems. In view of the presence of the TRW coating in the program, very few evaluation statements are presented concerning these data; rather the data is presented in its entirety for evaluation by the reader.

#### 4.1 Coatings

Consideration was given in the screening evaluation study to all of the presently available oxidation protective coatings applicable to columbium alloys. The following section lists the organizations which were contacted by letter and requested to participate in the program. This section also presents the information supplied by each coating organization regarding the coating process conditions used in preparing the evaluation specimens for the screening tests.

AMF - Evaluation specimens were supplied to American Machine and Foundry Corporation for application of the AMFkote No. 3 coating. However, the specimens were destroyed during the coating treatment due to a faulty temperature controller. Because of a long delivery schedule, time did not permit shipment of a second set of specimens for reapplication of this coating.

Boeing - The Boeing Company declined to participate in the program.

Chromalloy - All specimens were W-2 chromallized in Run Number XP-11321. No further information on processing was made available.

DuPont - DuPont Company declined to participate, indicating that their coating process was not yet sufficiently advanced for this type of evaluation.

General Electric - Due to heavy commitments on other programs, General Electric Company was unable to supply LB-2 coated evaluation specimens.

McDonnell - A request was made by McDonnell Aircraft Corporation to supply a set of evaluation specimens coated by the LB-2 aluminum slurry process. A 1/32" diameter hole was drilled in each specimen to provide a means of suspending the specimens during the coating application. Edges and corners of specimens were sanded where necessary with No. 1 emery paper. All specimens were vapor



degreased in trichloroethylene, pickled in an  $\text{HNO}_3$ -HF solution as per McDonnell Process Specification 13154, and LB-2 slurry coated and diffusion treated in an argon atmosphere retort according to the procedure of McDonnell Process Specification 13156. The average total thickness loss resulting from pickling was 0.0005". Micrometer measurements by McDonnell indicated an average coating thickness of 0.0026".

Pfautler - A Cr-Mo modified silicide coating was applied to the evaluation specimens by a two-cycle pack cementation process. No further information was made available.

Pratt & Whitney (Canel) - Specimens were coated utilizing the following procedure:

- 1) Wash in reagent grade acetone.
- 2) Pack in a mixture of 90 volume percent Micria "AD" alumina + 10 volume percent titanium and silicon powder in a one-to-one weight ratio. Anhydrous cupric chloride activator was added to the mixture in the ratio of 0.01 moles  $\text{CuCl}_2$  per mole of metal powder. The closed bomb was filled with argon and fired at 1800°F for 23 hours with a slow argon purge.
- 3) All pieces washed in cold water.
- 4) All pieces chromized using the same procedure as in (2) except that the pack mixture contained 10 volume percent of chromium powder and was fired at 1800°F for 16 hours.
- 5) All pieces washed in cold water.

GT&E - The specimens were originally supplied to Sylvania Electric Products Inc. a subsidiary of General Telephone and Electronics, Inc. for application of the Sylcor Al-Cr-Si alloy hot dipped coating. However, in view of the 2600°F screening test, which reportedly exceeds the maximum temperature at which the aluminide coating is protective, General Telephone and Electronics, Inc. elected to apply a silicide type coating. The coating was applied in a two-cycle pack process which consisted of a vacuum titanizing treatment followed by pack siliciding in an argon atmosphere.

TRW - The Cr-Ti-Si coating was applied by a three-cycle vacuum vapor deposition process. This coating process was developed and improved under Air Force contract. The evaluation specimens were

first precoated with titanium in a KF activated titanium pack for 6 hours at 1900°F. The second cycle consisted of forming a Cr-Ti alloy coating layer in a KF activated 50Cr-50Ti alloy pack in 8 hours at 2300°F. Silicon was then alloyed with the Cr-Ti layer for 4 hours at 2100°F in a KF activated silicon pack.

Vought - The coating applied to the evaluation specimens by Vought Aeronautics was basically a silicide coating containing intermetallic compounds of Cb, Cr, B and Fe, applied by a two-cycle pack cementation process. Silicon was deposited at 2100°F for 16 hours in the first cycle and Cr, B, and Fe at 2200°F for 16 hours in the second cycle. Each pack consisted of coating elements, a halide salt activator, and an inert filler material. The coating pack was contained in a slip coated steel retort equipped with a sand seal and operated in an air atmosphere at processing temperatures. Prior to coating all specimens were vapor blasted using pumice and approximately 40 psi pressure.

Therefore, only seven of the eleven organizations contacted supplied coated D-14 alloy specimens for the screening evaluation tests. The seven coatings included only one aluminide coating, applied by a slurry dip-diffusion process, and six basically silicide coatings applied by various pack type-vapor deposition techniques.

#### 4.2 Specimen Preparation

Sets of twenty-eight (28) evaluation specimens were prepared from the 30 mil D-14 alloy sheet and shipped to the various organizations for coating. The following preparation procedure was employed:

- 1) Specimens sheared to size -  
Oxidation coupons - 0.030" x 0.5" x 0.5"  
or 0.030" x 0.360" x 0.5"  
Bend specimens - 0.030" x 0.360" x 1.0"
- 2) All edges and corners rounded on a fine abrasive wheel
- 3) All specimens degreased in trichloroethylene
- 4) All specimens lightly etched in an aqueous HF-HNO<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub> solution.





The size of the oxidation coupon (No. 1 above) was reduced after several sets of specimens had been prepared due to a shortage of material. Each set of evaluation specimens contained fourteen (14) oxidation coupons and fourteen (14) bend specimens.

Any additional specimen preparation performed and reported by the coating organizations was indicated in the previous section (Section 4.1).

#### 4.3 Screening Test Results on Coated D-14 Alloy

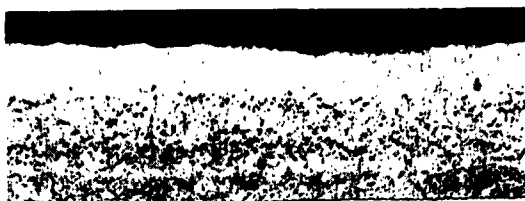
##### 4.3.1 Metallography

One specimen representing each of the seven coatings was metallographically prepared in the as coated condition. Figures 1 and 2 are photomicrographs showing the microstructures of each of these coatings on D-14 alloy, and the average coating thickness measurements are indicated beneath the corresponding pictures. The coatings ranged in thickness from 2 to 4 mils for the individual coatings. In general, the coatings were quite uniform over the specimen surfaces, with the exception of the GT&E coating.

Coating porosity appears to be quite abundant in the McDonnell LB-2 aluminide coating, as observed in the photomicrograph.

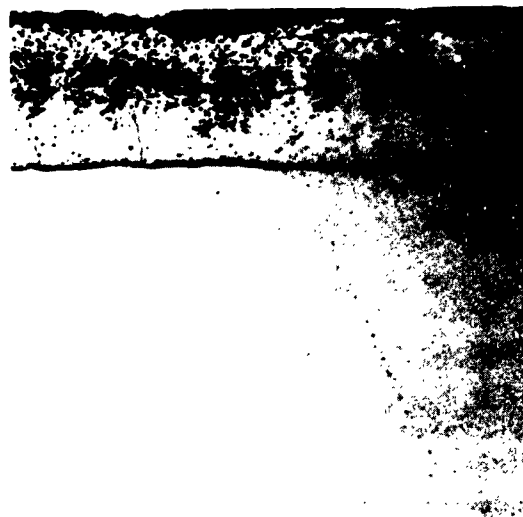
Pronounced differences can also be observed in the microstructures of the six modified silicide type coatings. For example, the chromium surface layer formed during the second coating cycle on the Pratt & Whitney coating, the pre-deposited titanium diffusion zone beneath the GT&E silicide coating and the intermetallic phase and titanium diffusion zone in the substrate beneath the TRW Cr-Ti-Si alloy coating resulting from the Cr-Ti coating cycle prior to the deposition of silicon.

A grain boundary precipitate in the D-14 alloy substrate is noted to a depth of 4-6 mils below the Pratt & Whitney coating, and to a lesser extent in the Pfaudler and Vought coated specimens. Microhardness traverses were conducted across specimens of each coating to investigate the possibility of interstitial contamination during the coating process. These data are listed in Table 2. There was no evidence of substrate hardening resulting from the observable contamination. The variation in the as coated substrate core hardness was basically a function of residual cold work. Since the coating application treatments ranged in temperature from 1800-2300°F, varying degrees of stress relief and recrystallization were experienced by the wrought alloy sheet.



Chromalloy

3.4 Mils



McDonnell

3.0 Mils



Pfaudler

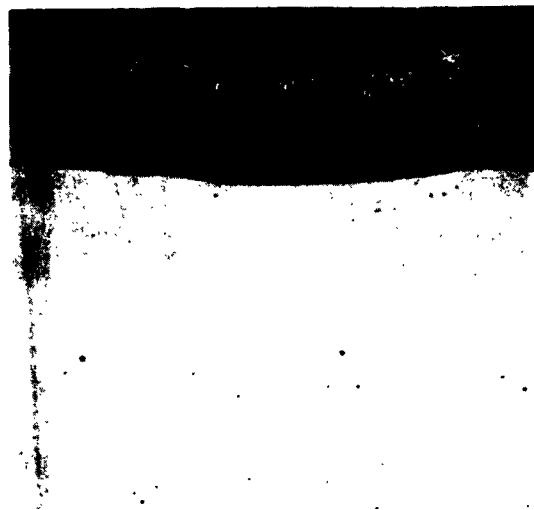
2.6 Mils



Pratt & Whitney

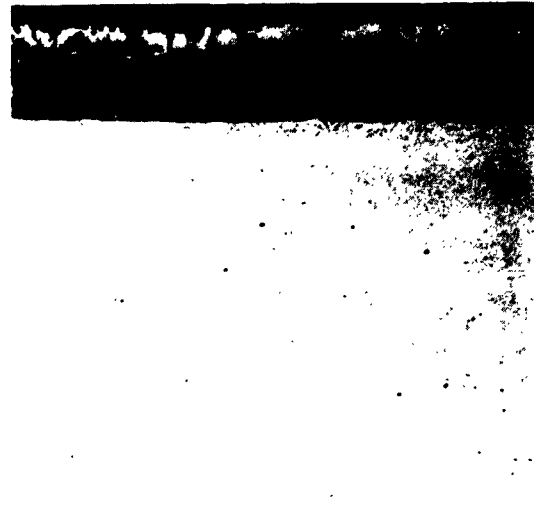
2.0 Mils

Figure 1 Photomicrographs and Average Coating Thickness Measurements  
of Various Coatings on D-14 Alloy in the As Coated Condition  
250X



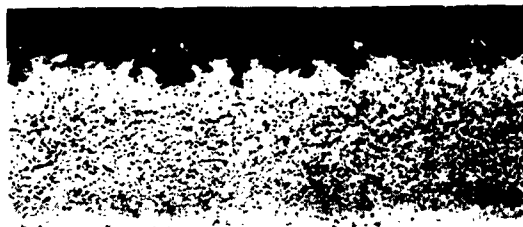
GT&E

2.8 Mils



TRW

2.4 Mils



Vought

4.0 Mils

Figure 2 Photomicrographs and Average Coating Thickness Measurements  
of Various Coatings on D-114 Alloy in the As Coated Condition  
250X

TABLE 2

Microhardness Data On As Coated 30 Mil D-14 Alloy  
Coated By Various Organizations

<u>Coating Organization</u>	<u>Microhardness Traverse</u>	
	<u>Distance from Surface</u> <u>0.001"</u>	<u>D.P.H. Average Core</u> <u>Hardness -D.P.H.</u>
Chromalloy	1.7	1412
	3.4*	-
	4.0	200
	7.0	187
	11.0	187
	15.0	187
	19.0	187
McDonnell	1.2	240
	2.5*	-
	3.5	180
	7.0	160
	11.0	168
	15.0	170
	19.0	159
Pfaudler	1.5	645
	3.0*	-
	4.0	152
	7.0	131
	11.0	140
	15.0	152
	19.0	145
PWA	1.0	1043
	2.0*	-
	3.0	216
	6.0	145
	10.0	155
	14.0	143
	18.0	145

\* Distance of the coating-base metal interface from the coating surface



TABLE 2 (CONTINUED)

<u>Coating Organization</u>	<u>Microhardness Traverse</u>		
	<u>Distance from Surface</u> <u>0.001"</u>	<u>D.P.H.</u>	<u>Average Core</u> <u>Hardness-D.P.H.</u>
GT&E	1.5	841	
	3.0*	-	
	4.0	307	
	7.0	130	131
	11.0	130	
	15.0	131	
	19.0	134	
TRW	1.0	732	
	2.0*	-	
	3.0	208	
	6.0	134	135
	10.0	138	
	14.0	134	
	18.0	136	
Vought	2.0	1302	
	4.0*	-	
	5.0	175	
	8.0	150	154
	12.0	163	
	16.0	150	
	20.0	155	

\* Distance of the coating-base metal interface from the coating surface

#### 4.3.2 Cyclic Oxidation

Cyclic oxidation tests were conducted in air at 1600, 2000, 2300 and 2600°F in globar heated box type furnaces. Three specimens representing each coating were tested at each temperature. At 2300 and 2600°F the specimens were placed on individual aluminum oxide pads in a large platinum pan which was placed on a refractory pedestal in the furnace hearth. At 1600 and 2000°F the specimens were placed on aluminum oxide pads in Inconel boats.

Temperature control in the oxidation furnaces was maintained with Pt-Pt+10%Rh thermocouples adjacent to the furnace heating elements. The specimen temperatures were monitored by second thermocouples in contact with the specimen boats.

The testing sequence at 1600, 2000, and 2300°F involved cycling the specimens to room temperature once each hour for observation during the first 24 hours of testing. Thereafter the specimens were exposed for 16 hours followed by 8 hours of cyclic oxidation in each succeeding 24 hour period. All specimens were exposed until coating failure, or for 150 hours, at which time the tests were terminated. At 2600°F the specimens were cycled to room temperature once each hour throughout the test sequence until coating failure occurred. Failure time was defined as the point of first positive observation of a coating defect and the growth of columbium oxide through the coating at any location on the specimen.

Figure 3 is a photograph of the specimens oxidized under cyclic conditions at 1600°F. The time to failure or the test termination time of 150 hours is shown for each specimen. These oxidation test data are also tabulated in Table 3. With the exception of the McDonnell LB-2 coating, almost all coating failures were initiated at specimen edges or corners at 1600°F. The Pratt & Whitney and TRW coatings were the only coatings which successfully protected all three D-14 alloy specimens (100% reliability) for the 150 hour exposure at 1600°F.

Figure 4 is a photograph of the coupons oxidation tested at 2000°F. These data are also tabulated in Table 3. Again the coating failures were more prevalent at specimen edges and corners where coatings are inherently less reliable. The majority of the coatings were incapable of protecting the D-14 alloy for even 50 hours at 2000°F. Only the TRW coating protected all three specimens for 150 hours at 2000°F.



1600°F

Chromalloy			McDonnell			Pfautler			Pratt & Whitney		
4	150	150	4	4	5	67	150	150	150	150	150
Hours			Hours			Hours			Hours		
Chromalloy			McDonnell			Pfautler			Pratt & Whitney		
5	64	62	180	150	150	1	100	150			
Hours			Hours			Hours			Weight		

Figure 3 Coated D-14 Alloy Specimens Cyclic Oxidized in Air Until Coating Failure or for 150 Hours at 1600°F

TABLE 3

Protective Life Of Various Coatings On D-14 Alloy  
Cyclic Oxidation Tested In Air At 1600, 2000, 2300 & 2600°F

Coating Organization	Protective Life - Hours(1)			
	1600°F	2000°F	2300°F	2600°F
Chromalloy	4, 150, 150	5, 18, 43	5, 6, 16	1, 1, 9
McDonnell	4, 4, 5	16, 16, 17	5, 5, 16	2, 2, 2
Pfandler	67, 150, 150	5, 66, 69	1, 1, 17	9, 11, 11
Pratt & Whitney	150, 150, 150	17, 39, 44	3, 3, 19	1, 2, 2
GT&E	5, 61, 82	6, 21, 39	43, 62, 150	17, 19, 24
TRW	150, 150, 150	150, 150, 150	150, 150, 150	21, 27, 37
Vought	1, 150, 150	130, 150, 150	17, 18, 18	6, 13, 13

(1) Oxidation exposure terminated at 150 hours at 1600, 2000 and 2300°F



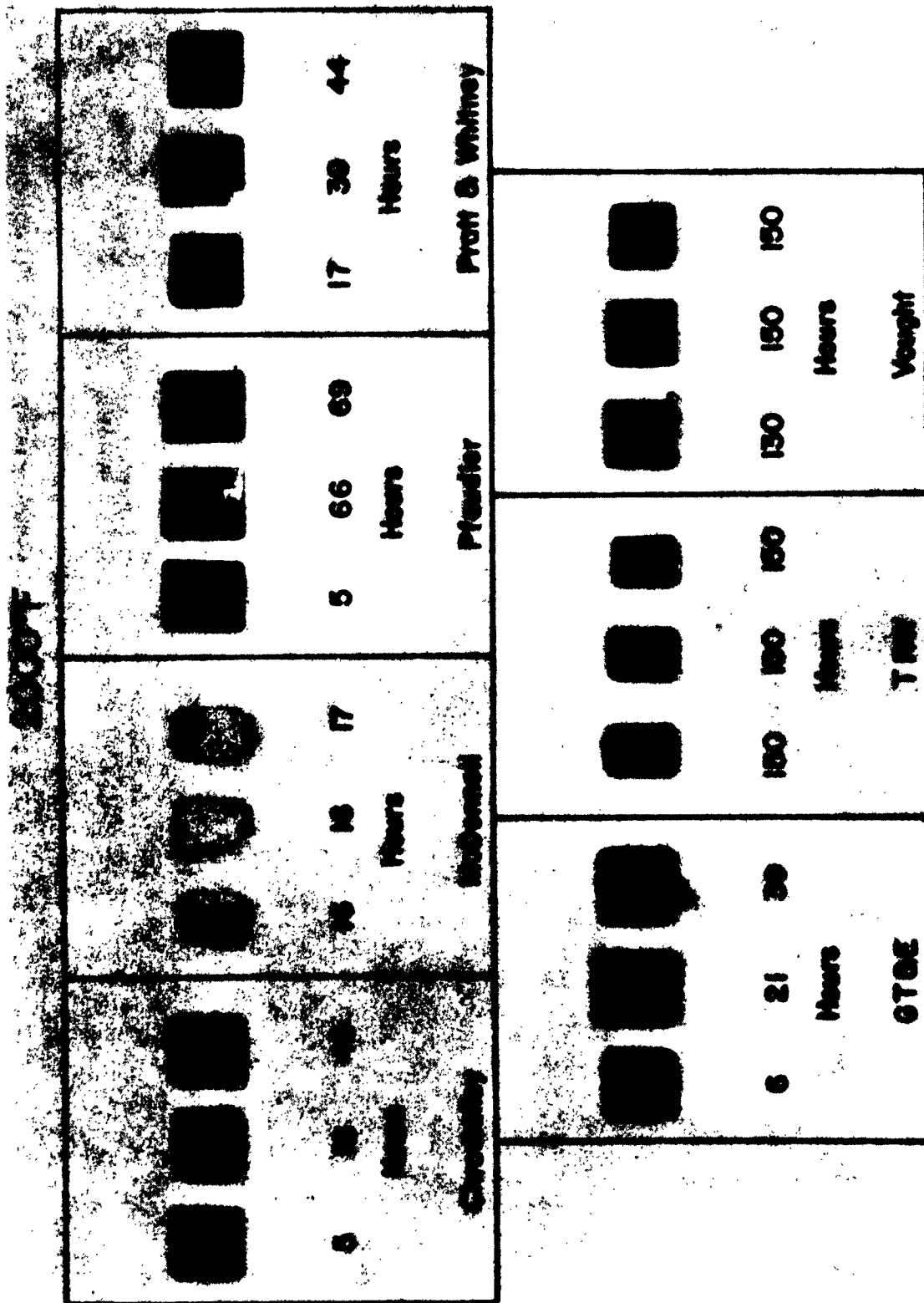


Figure 4 Coated D-114 Alloy Specimens Cyclic Oxidized in Air Until Coating Failure or for 150 Hours at 2000°F

LX

Figure 5 is a photograph showing the group of specimens cyclic oxidation tested at 2300°F. These data are also listed in Table 3. Coating failures occurred on the majority of specimens in less than 20 hours at 2300°F. Only the TRW coating protected all three specimens for 150 hours of exposure.

The group of specimens oxidation tested at 2600°F are shown in Figure 6 and the data are tabulated in Table 3. All of the coatings tested failed in less than 40 hours at 2600°F with only the GT&E and TRW coatings providing protection for times in excess of 20 hours. The LB-2 coating failed catastrophically in less than 2 hours, forming a corrosive slag which completely destroyed the specimens. A visible glassy phase formed on the surface of all coatings exposed at 2600°F, with the exception of the Pratt & Whitney coating.

Although several specimens were cyclic oxidation tested for 150 hours without external evidence of coating failure, this was no assurance the coatings had not leaked oxygen and/or nitrogen in sufficient quantity to embrittle the substrate. Representative specimens were therefore sectioned and microhardness traverses were made across the metallographically prepared cross sections. Photomicrographs of Pfadler, Pratt & Whitney and Vought coated D-14 alloy specimens which had not failed in 150 hours at 1600 and 2000°F are shown in Figure 7, including the microhardness impressions and Diamond Pyramid Hardness (DPH) values (100 gram load). Figure 8 shows similar photomicrographs of TRW coated specimens which had not failed in 150 hours at 1600, 2000 and 2300°F.

All coated specimens oxidized for 150 hours without failure at 1600-2300°F indicated softening of the substrate as a result of more thorough annealing during the long time exposure. Many of the oxidized coated specimens did show evidence of intergranular substrate contamination, however, no significant hardness increase of the base metal was registered. Apparently the limited amount of contamination, resulting from the slow diffusion of oxygen and/or nitrogen through the coatings, was tied up by zirconium in the alloy and confined primarily to precipitation in the grain boundaries with little or no interstitial hardening of the substrate.

An alternative method of revealing sub-surface contamination, that of oxidation exposure followed by mechanically testing to investigate substrate embrittlement, will be discussed in Section 4.3.4.



2300°F

Chromalloy			McDonnell			Pfautler			Pratt & Whitney		
5	6	16	5	5	16	1	1	17	3	3	19
Hours			Hours			Hours			Hours		
Chromalloy			McDonnell			Pfautler			Pratt & Whitney		
43	62	150	150	150	150	150	150	150	17	18	18
Hours			Hours			Hours			Hours		
GT&E			TRW			Vought			Vought		

Figure 5 Coated D-114 Alloy Specimens Cyclic Oxidized in Air Until Coating Failure or for 150 Hours at 2300°F

LI

2600°F








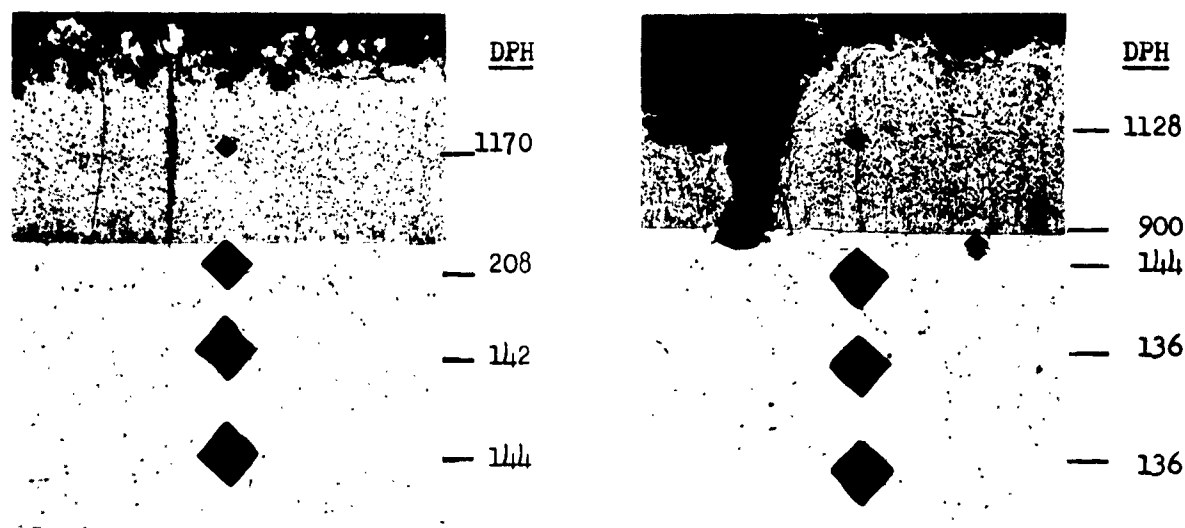
Chromalloy				McDonnell				Pfaudler				Pratt & Whitney			
															
1	1	9	Hours	2	2	2	Hours	9	11	12	Hours	1	2	2	Hours
GT&E				TRW				Vought							
															
17	19	24	Hours	21	27	37	Hours	6	13	13	Hours				

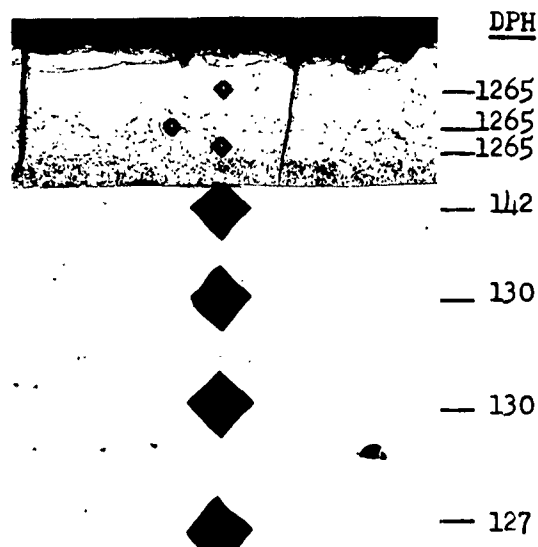
Figure 6 Coated D-11 Alloy Specimens Cyclic Oxidized in Air Until Coating Failure at 2600°F 1X



150 Hours - 1600°F

150 Hours - 2000°F

Vought



150 Hours - 1600°F

Pfautler

Figure 7 Photomicrographs of Vought and Pfautler Coated D-11 Alloy Specimens Oxidized for 150 Hours at 1600 and 2000°F, Showing Microhardness Impressions 250X



— 129

← 127

← 132

150 Hours - 1600°F

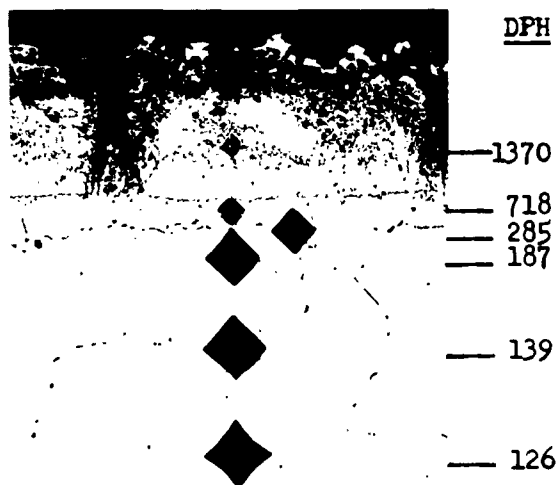


— 147

— 136

— 138

150 Hours - 2000°F



— 139

— 126

150 Hours - 2300°F

TRW

Figure 8 Photomicrographs of TRW Cr-Ti-Si Coated D-14 Alloy Specimens Oxidized for 150 Hours at 1600, 2000, and 2300°F, Showing Microhardness Impressions 250X



#### 4.3.3 Room Temperature Bend Ductility Tests on As Coated Specimens

Room temperature bend tests were conducted on as coated specimens of each coating system in order to determine the influence of the coating and coating process conditions on the room temperature ductility of the D-14 alloy sheet. The bend tests were conducted over a 4T radius at a crosshead speed of 10 inches per minute, in accordance with specifications recommended by the Refractory Metal Sheet Rolling Panel of the Materials Advisory Board. For 30 mil sheet this involved the following parameters:

- 1) Specimen width - 12T - 0.360"
- 2) Bend radius - 4T - 0.120"
- 3) Beam support distance - 15T - 0.450"
- 4) Crosshead speed - 10 inches/minute

Table 4 lists the bend test results for the as coated D-14 alloy sheet. The bend transition temperature of the uncoated sheet was -320°F after a one hour stress relief at 1800°F and after a ten hour vacuum anneal at 2600°F. The Chromalloy, Pratt & Whitney and Vought coating treatments raised the bend transition temperature of the D-14 alloy sheet above room temperature. A 90° bend was obtained with one Pratt & Whitney specimen, however, cracks were produced in the bend cross section indicating a transition temperature near room temperature. All of the other coated specimens gave 90° bends at room temperature. Thus three of the seven coatings evaluated embrittled the D-14 substrate for deformation at the normal handling temperature. The substrate embrittlement could have resulted from either oxygen or nitrogen contamination during the coating treatment, or from the use of hydrogen as a process carrier gas.

#### 4.3.4 Cyclic Oxidation + Room Temperature Bend Ductility Tests

With a relative baseline ductility established for D-14 alloy sheet in the as coated condition, coated specimens were then exposed in an oxidizing atmosphere under cyclic conditions for 10 hours at 2600°F and for 50 hours at 2000°F, and subsequently bend tested at room temperature. These specimens were exposed in conjunction with the oxidation test coupons discussed in Section 4.3.1. Coatings which were poor barriers to the diffusion of oxygen could permit severe embrittlement and hardening of the coated substrate prior to any external evidence of coating failure.

TABLE 4

Bend Test Results For Various Coatings On 30 Mil D-14 Alloy  
As Coated And After Oxidation For 50 Hours At 2000°F And  
10 Hours At 2600°F In Air

Coating Organization	As Coated	Bend Angle - Degrees		
		Preoxidized - 2000°F(a)	Preoxidized - 2600°F(a)	Preoxidized - 2600°F(a)
Chromalloy	0 (c)	90	90	90 (8)* 90 90
McDonnell	90	0 (16)	0 (16)	(b) (2) (b) (2) (b) (2)
Pfaudler	90	90	90	(b) (7) 90* 90*
Pratt & Whitney	90* (d)	90	90*	0 (1) 90 (1)* 90 (4)*
GT&E	90	90 (39)*	90	0 90* 90*
TRW	90	90	90	90 90 90
Vought	0	90	90	90 90* 90

a) Preoxidation 50 hours at 2000°F and 10 hours at 2600°F except  
for times noted in parenthesis

b) Specimens melted during preoxidation - no bend test

c) Specimen not returned

d) \* Denotes base metal cracked partially through bend cross section





Baseline data for the uncoated Cb-5Zr alloy sheet (30 mil) was first established for these screening tests by bend testing uncoated D-14 alloy sheet in the stress relieved condition (1 hour at 1800°F in vacuum), after 10 hours vacuum heat treatment at 2600°F, and after short time exposure in air at 2000 and 2600°F. The bend tests were conducted over the temperature range -320 to 1200°F. Figure 9 shows a plot of the permanent bend angle as a function of test temperature for each of these conditions. These data are also listed in Table 5.

The D-14 alloy was ductile at -320°F both in the stress relieved condition (1 hour at 1800°F) and after 10 hours vacuum heat treatment at 2600°F. Figure 10 shows photomicrographs illustrating the degree of grain growth resulting from the 2600°F heat treatment.

The severity of the extremely short time oxidation exposure on the uncoated columbium alloy at 2000 and 2600°F is evidenced by the rapid increase of the ductile to brittle transition temperature (Figure 9). Oxidation for 0.1 hour at 2000°F raised the transition temperature from below -320°F to approximately -100°F, after 0.3 hour to 1100°F, and after 0.5 hour at 2000°F the 30 mil Cb-5Zr alloy sheet was brittle above 1200°F. Oxidation for 0.1 hour at 2600°F rendered the 30 mil sheet brittle above 1200°F.

Figure 11 presents photomicrographs of the specimens oxidized at 2000°F. The gross internal penetration of oxygen is observed as the advancing interface of oxygen contaminated substrate across the cross section. Microhardness impressions are shown in the photomicrographs and the hardness values are plotted as a function of distance from the original surface in Figure 12. At 2000°F the D-14 alloy was hardened by oxygen to a depth of 15 mils, completely through the 30 mil sheet, in 0.5 hour. At 2600°F the entire specimen cross section was severely hardened in only 0.1 hour of oxidation.

Figure 13 is a plot of internal oxygen contamination and cross sectional surface recession as a function of time at 2000 and 2600°F. Internal penetration was taken as the depth of the metallographically observed interface which was produced on polishing by the differential hardness. This interface was not quite coincident with the depth of contamination indicated by the microhardness traverses. Surface recession, resulting from conversion of the base metal to an oxide scale, increased by a factor of approximately 10 from 2000 to 2600°F.

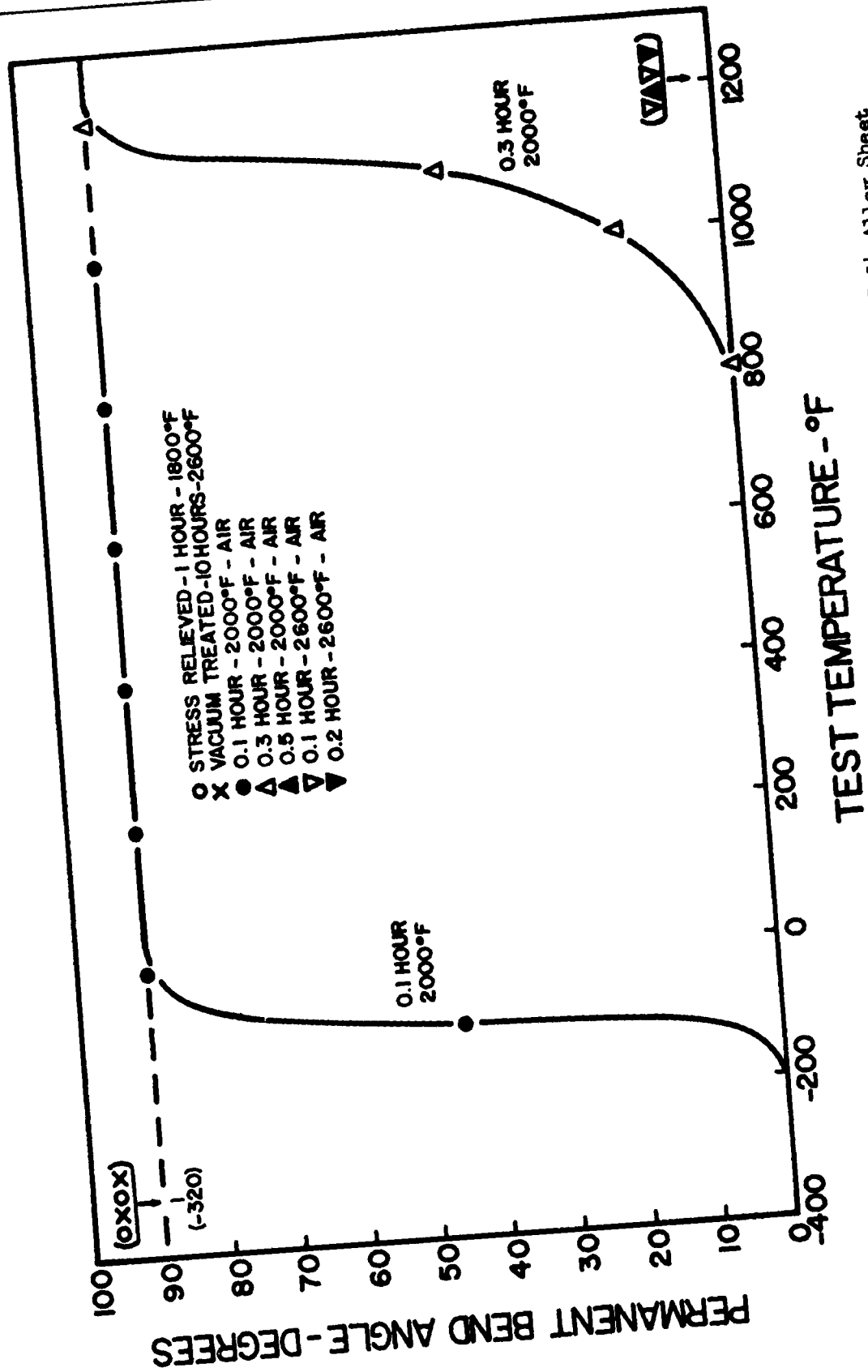


Figure 9 Bend Test Data for Vacuum Heat Treated and Air Oxidized 30 Mil D-14 Alloy Sheet

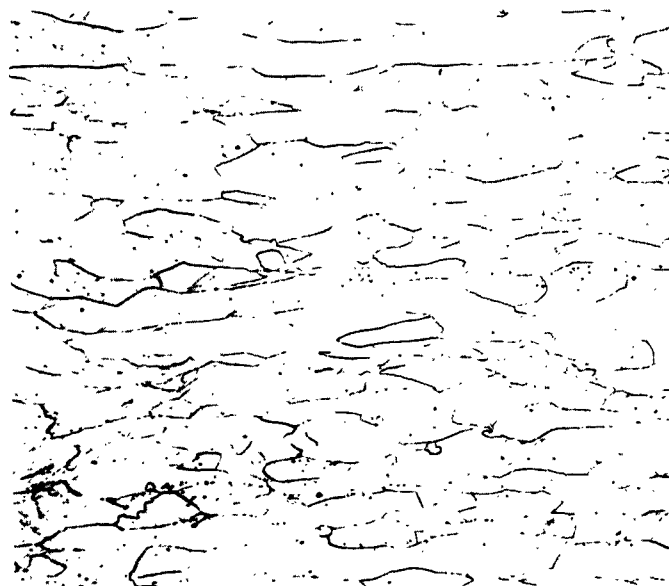


TABLE 5

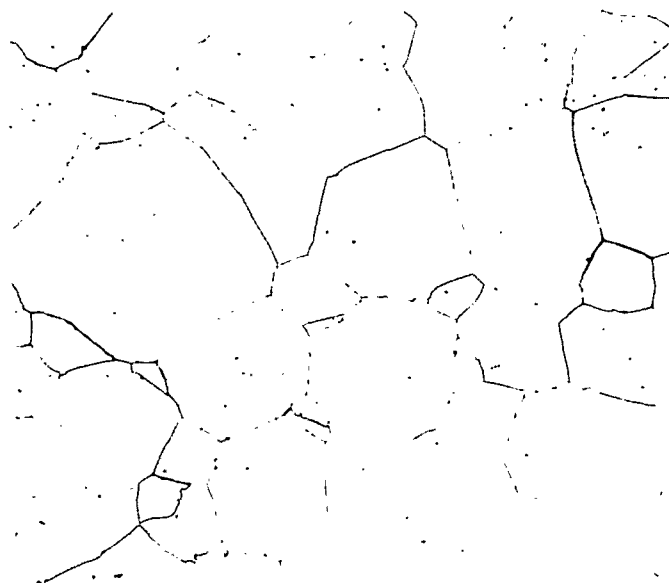
Bend Ductility Tests On 30 Mil D-14 Alloy Sheet In Various Conditions At Various Temperatures(1)

Specimen Condition	Degrees Bend (4T Radius)									
	-320	-90	R.T.	200	400	600	800	1000	1100	1200
Stress Relieved	90, 90									
Vacuum Treated-										
10 Hours-2600°F	90, 90									
Oxidized-										
0.1 Hour-2000°F		45	90	90	90	90	90	90		
0.3 Hour-2000°F								15	40	90
0.5 Hour-2000°F							0	0		0, 0
0.1 Hour-2600°F										0, 0
0.2 Hour-2600°F										0, 0

(1) Tests conducted according to MAB specifications

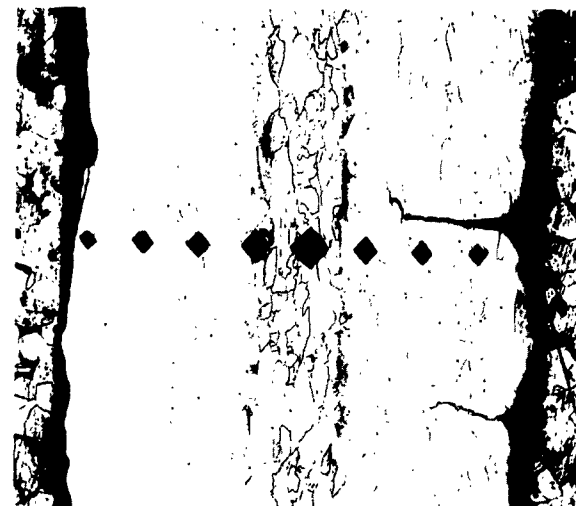


1 Hour - 1800°F

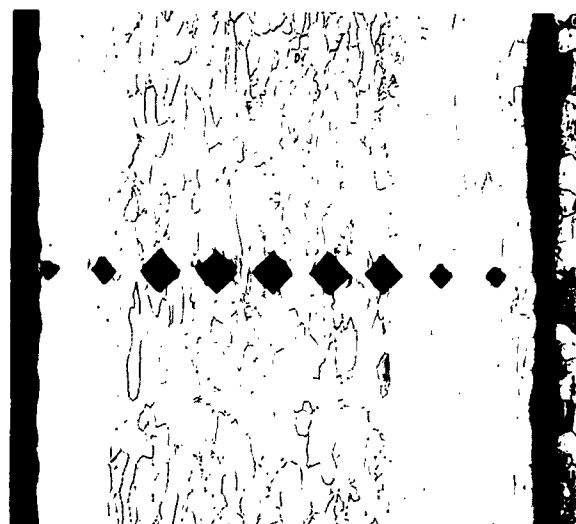


10 Hours - 2600°F

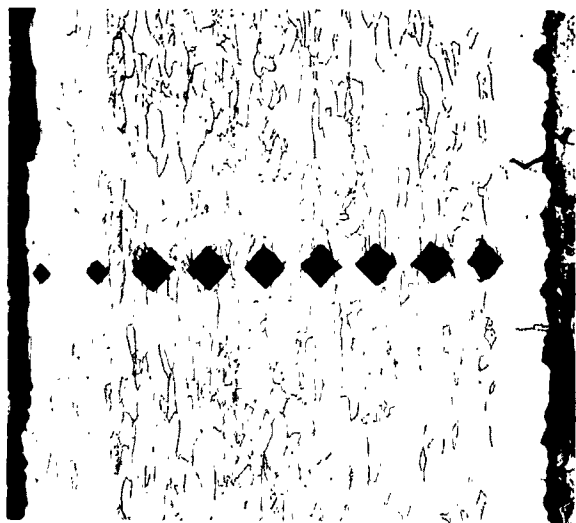
Figure 10 Photomicrographs Showing D-11 Alloy Sheet  
Structure as Stress Relieved (1 Hour - 1800°F)  
and After Vacuum Heat Treatment for 10 Hours  
at 2600°F 250X



0.5 Hour



0.3 Hour



0.1 Hour

Figure 11 Photomicrographs Showing Oxygen Penetration in 30 Mil  
D-114 Alloy Sheet Oxidized in Air at 2000°F 100X

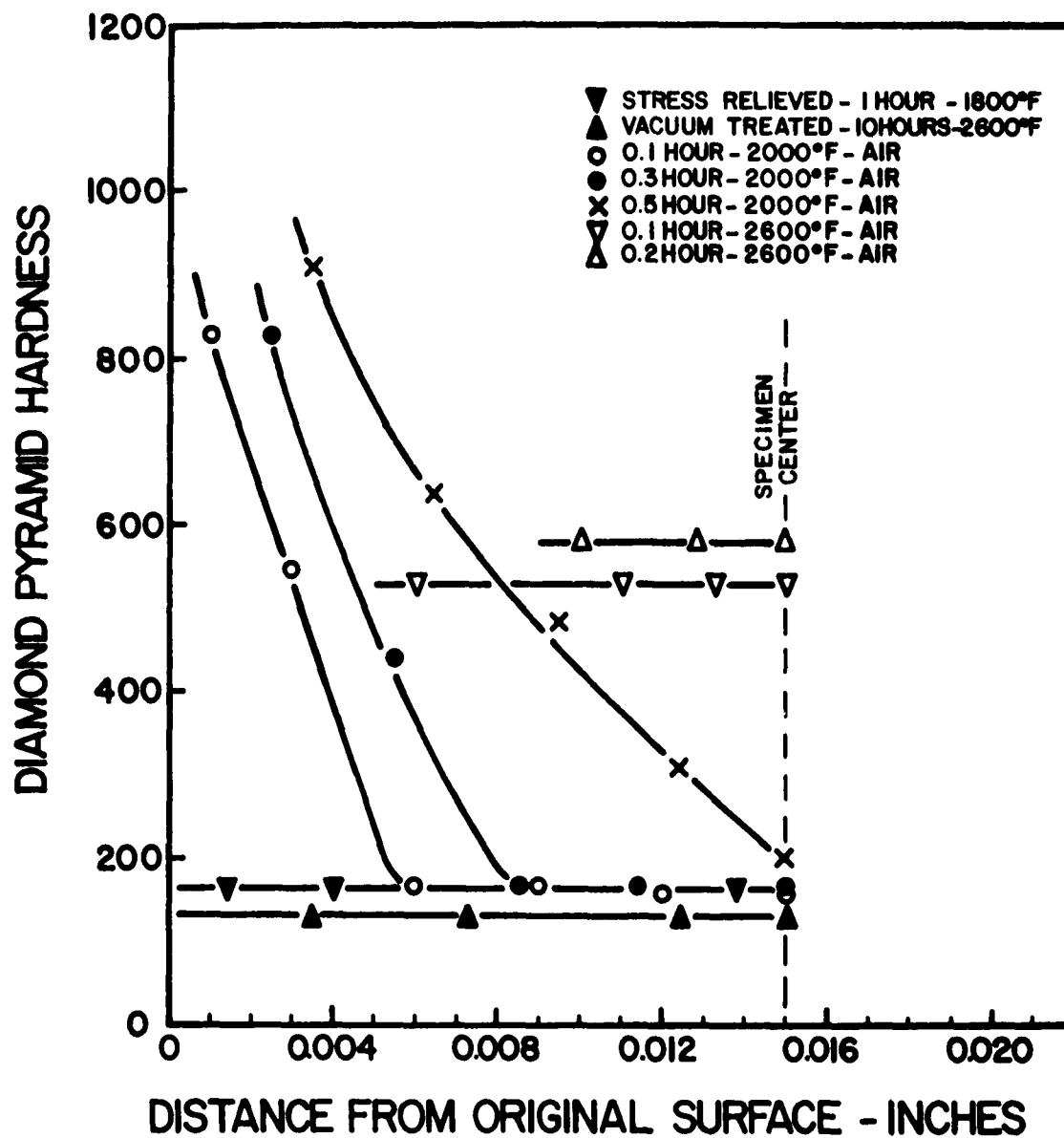


Figure 12 Microhardness Traverses Across 30 Mil D-11 Alloy Sheet for Heat Treated and Oxidized Conditions

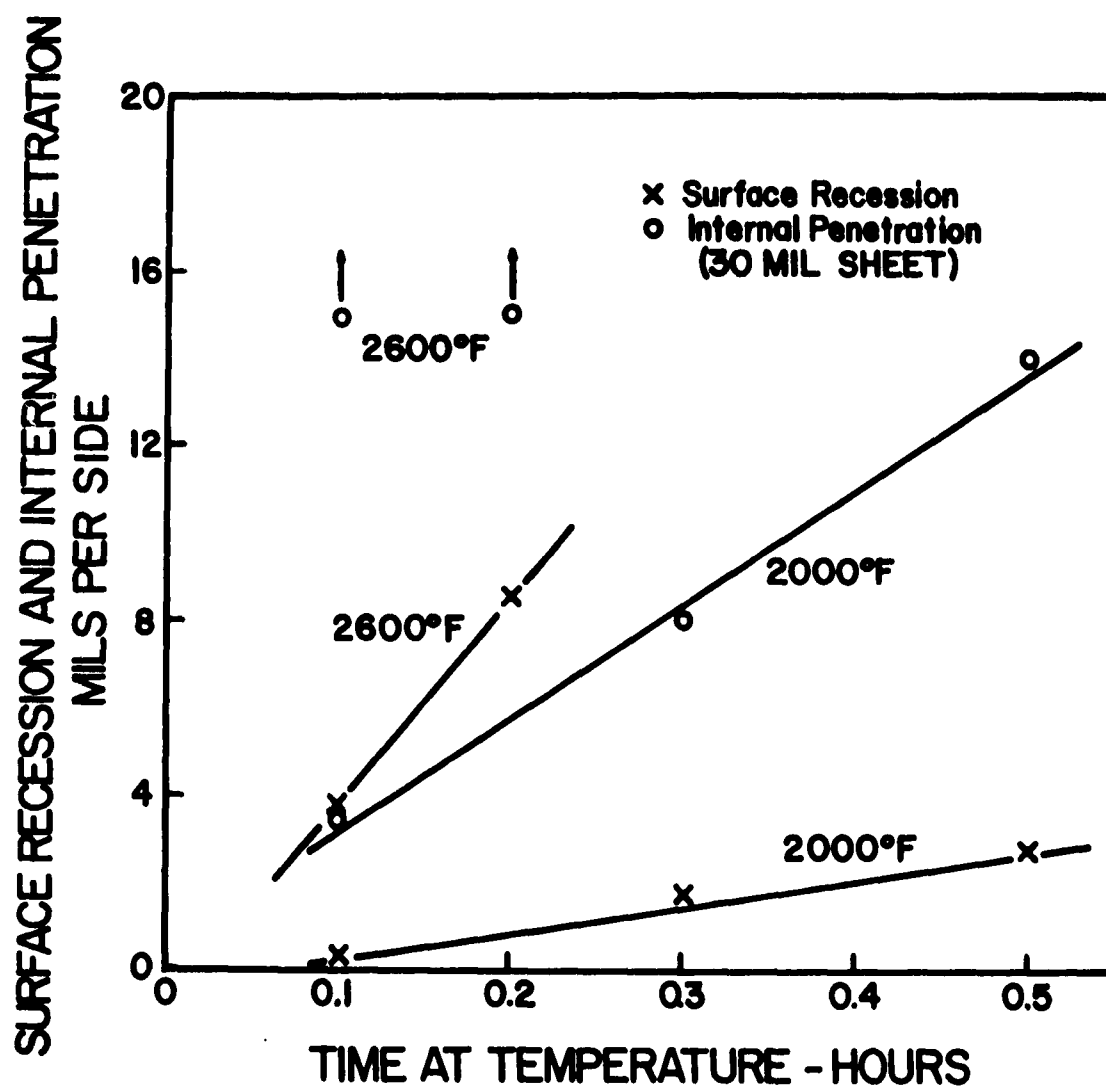


Figure 13 Surface Recession and Internal Penetration for Oxidized D-14 Alloy as a Function of Time at 2000 and 2600°F

A comparison can now be made of the bend transition temperature with the photomicrographs, microhardness traverses, and measurements of oxygen affected metal. Oxidation of the unprotected 30 mil D-14 alloy sheet for 0.1 hour at 2000°F contaminated approximately 25% of the original cross section, raising the transition temperature from below -320°F to -100°F. In 0.3 hour at 2000°F 75% of the cross section was affected by oxygen and the transition temperature was increased to 1100°F. Oxygen completely penetrated the 30 mil sheet in 0.5 hour at 2000°F and 0.1 hour at 2600°F, embrittling the metal, as measured in the bend test, to above 1200°F. With these data as a descriptive evaluation of the sensitivity of D-14 alloy and other columbium alloys to interstitial embrittlement, the effectiveness of these coatings in preventing this embrittlement can be appreciated.

Three coated D-14 alloy bend specimens representing each of the seven different coatings were cyclic oxidation tested for 50 hours at 2000°F and 10 hours at 2600°F, in conjunction with the oxidation test coupons discussed previously, and subsequently bend tested at room temperature. Again the bend tests were conducted according to the MAB specifications. As pointed out previously, it was the intention of these tests to point out those coatings which were relatively poor barriers to the diffusion of oxygen.

Figure 14 is a photograph of the specimens oxidized for 50 hours at 2000°F and then bend tested at room temperature. The bend test results are listed in Table 4 (page 25). The exposure at 2000°F was originally intended to be 100 hours, however, in view of the numerous coating failures at relatively short times the oxidation exposure was terminated after 50 hours.

One Chromalloy specimen failed at a corner after 5 hours, however, remained in the test for the 50 hour exposure. All three Chromalloy specimens gave a 90° bend, indicating a recovery from the room temperature embrittlement caused by the coating treatment.

The McDonnell LB-2 coated specimens failed and were removed from the furnace after 16 hours at 2000°F. All three specimens were brittle at room temperature.

Failures were observed outside the bend section on the Pratt & Whitney specimens after 16, 23 and 39 hours. However, the oxidation was continued for 50 hours and the specimens were ductile in the bend section at room temperature.





2000°F





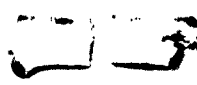
















Chromalloy			McDonnell			Pfadtler			Pratt & Whitney		
  			  			  			  		
50	50	50	16	16	16	50	50	50	50	50	50
Hours			Hours			Hours			Hours		
Chromalloy			McDonnell			Pfadtler			Pratt & Whitney		
GT&E			TRW			Vought					
  			  			  					
39	50	50	50	50	50	50	50	50			
Hours			Hours			Hours					

Figure 14 Room Temperature Bend Tested Coated D-14 Alloy Specimens Cyclic Oxidation Tested a  
Maximum of 50 Hours at 2000°F in Air Prior to Bending LX

GT&E coated specimens failed locally at 16 and 23 hours, and one specimen was removed after 39 hours as shown in the photograph. Both specimens which had failed in less than 39 hours were embrittled, however, the third specimen gave a 90° room temperature bend without cracking.

Only the Pfaudler, TRW and Vought coatings protected all three specimens from both localized failure and embrittlement for 50 hours at 2000°F.

Figure 15 is a photograph of the bend test specimens oxidized at 2600°F and room temperature bend tested. The bend test results are listed in Table 4. The oxidation tests at 2600°F were terminated at 10 hours.

Only the TRW coating protected the D-14 alloy against both localized coating failure and substrate embrittlement for 10 hours at 2600°F.

The Chromalloy coated specimens showed localized coating failures after 5 hours, however, two specimens were ductile at room temperature after the 10 hour exposure. Cracks occurred near the coating failure on bending the third specimen which was removed after 8 hours at 2600°F.

All of the McDonnell LB-2 coated specimens failed after 2 hours at 2600°F and were destroyed by a molten corrosive oxide slag.

One Pfaudler specimen failed and melted after 7 hours at 2600°F. The remaining two specimens showed localized failures at 5 hours, but were exposed for 10 hours. Bend angles of 90° were obtained at room temperature, however, cracks were produced in the bend cross section in both specimens.

Pratt & Whitney coated specimens failed and were removed after 1, 1 and 4 hours as shown. One specimen was brittle and two cracked severely although bending 90° at room temperature without complete fracture.

The Vought coated specimens showed localized coating failures after 6 hours at 2600°F, but were continued through the 10 hour exposure. One specimen cracked on bending near a localized failure, however, all three specimens gave 90° bends at room temperature.

No external evidence of coating failure was observed on the GT&E coated specimens after 10 hours at 2600°F, however, one

2600°F

























































Chromalloy			McDonnell			Pfadtler			Pratt & Whitney		
											
8	10	Hours	2	2	Hours	7	10	Hours	1	1	Hours
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			
											
	10	Hours					10	Hours			

Figure 15 Room Temperature Bend Tested Coated D-11 Alloy Specimens Cyclic Oxidation Tested a  
Maximum of 10 Hours at 2600°F in Air Prior to Bending  
IX

specimen was brittle and two cracked, although bending 90° when bend tested at room temperature.

Thus, only in the case of the GT&E coating was there substrate embrittlement without external evidence of coating failure. Figure 16 shows photomicrographs of a GT&E coated bend specimen embrittled after 50 hours oxidation at 2000°F and an oxidation coupon cyclic oxidized without external failure for 150 hours at 2300°F. Sufficient substrate contamination occurred in 50 hours at 2000°F to raise the bend transition temperature of the GT&E coated D-14 substrate above room temperature, even though the microhardness traverse indicates a soft matrix with a heavy oxide precipitate in the grain boundaries. The intergranular and dispersed oxide phase in the substrate of the specimen oxidized at 2300°F further indicates that the GT&E silicide coating was not impervious to the diffusion of oxygen at these temperatures. Gettering of the oxygen by the zirconium in the alloy and the precipitation of an intergranular oxide phase accounts for the embrittlement of the substrate without the evidence of hardening of the matrix.

#### 4.3.5 Room Temperature Prestrain + Cyclic Oxidation

In addition to the characteristic protective nature of a coating and the influence of the coating treatment on the low temperature properties of a coated substrate, an equally important property of a coating-base metal system is its tolerance for elastic or plastic deformation without loss of protective characteristics. Specimens of each coating were prestrained at room temperature to three different deformation levels. One level involved only base metal elastic strain and the other two involved elastic + different degrees of base metal plastic strain. The coated bend test specimens were deflected at room temperature to bend angles of 2, 5 and 10 degrees in the bend test fixture utilized in the room temperature bend testing (Sections 4.3.3 and 4.3.4). The Instron testing machine cross head speed was 0.020 inches per minute. Figure 17 is a representative load-deflection plot for the D-14 alloy sheet in the stress relieved and annealed conditions. Bend angles of 2, 5 and 10 degrees were selected from this plot to represent bending strains in the regions of both elastic and elastic + plastic deformation. All specimens were examined at low magnification (20X) following prestraining and in no case were coating cracks evident on specimens deflected to angles of 2 and 5 degrees, even though permanent deformation was visible after the 5 degree deflection. However, coating cracks were observed on all specimens deflected 10 degrees, and in most cases the coatings spalled at the specimen edges on the bend axis where the more severe

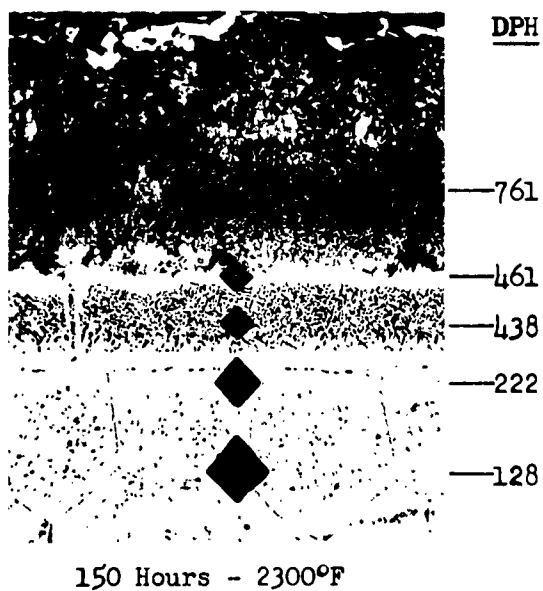
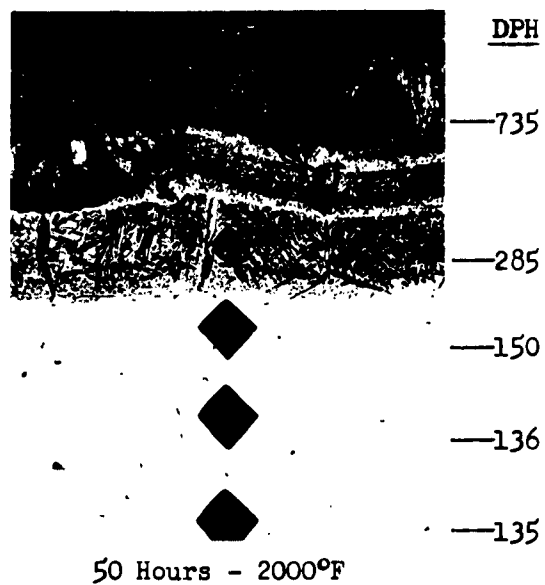


Figure 16 Photomicrographs of GT&E Coated D-14 Alloy Specimens Oxidized at 2000 and 2300°F, Showing Microhardness Impressions 250X

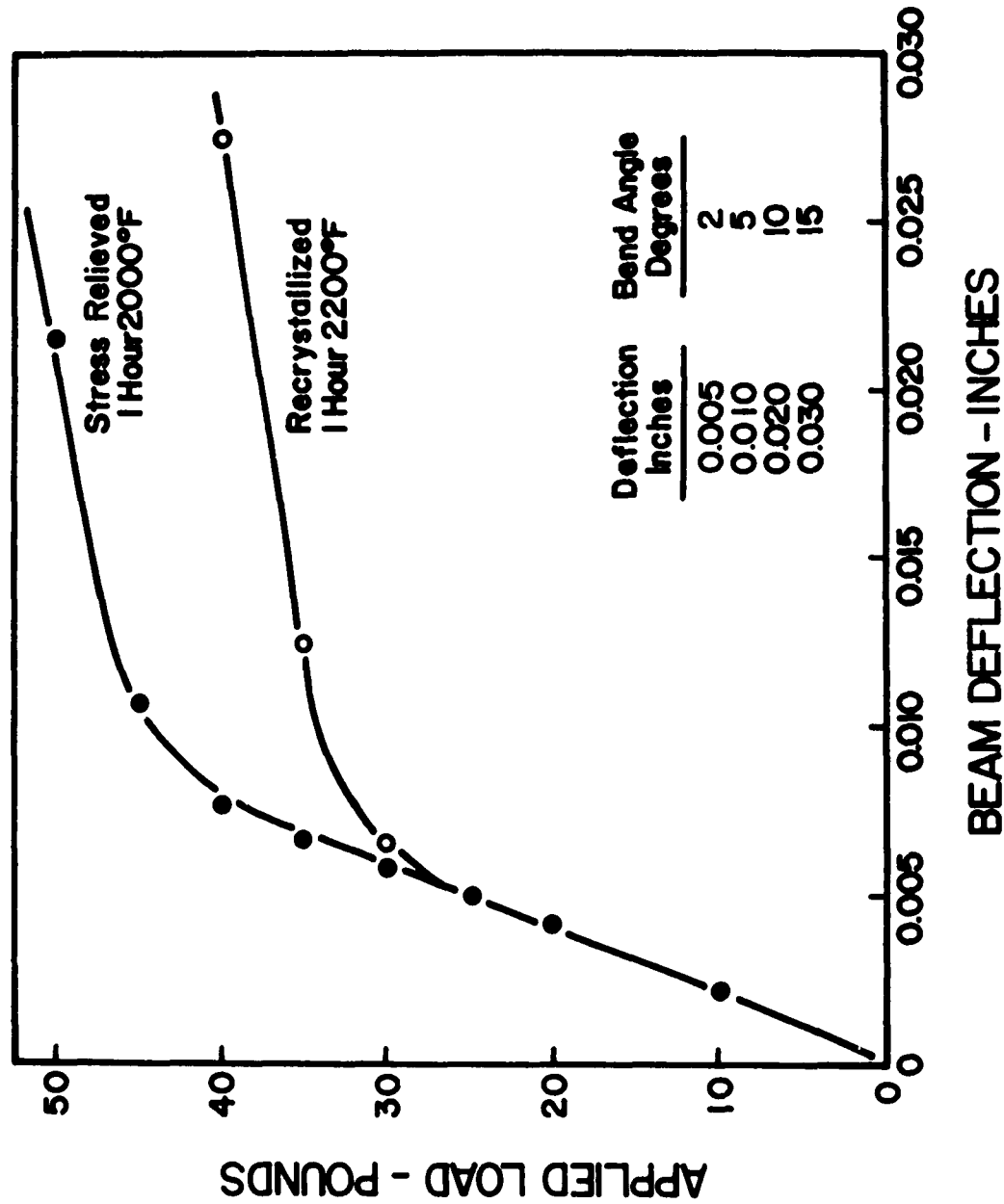


Figure 17 Beam Deflection as a Function of Applied Load for  
D-114 Alloy Stressed in the Bend Test Apparatus



strain conditions were encountered.

Following bending, specimens at each deformation level were cyclic oxidation tested at 2000°F for 10 hours and at 2600°F for 2 hours. In this way the protective characteristics of the coatings were observed at temperatures both below and within the range of reported coating self-healing mechanisms. All specimens were exposed for the 10 hour and 2 hour limits at 2000 and 2600°F respectively, regardless of the initiation of localized coating failure, in order to comparatively evaluate the relative degree of damage imposed by the room temperature prestrain for a subsequent uniform exposure at the two test temperatures. With the exception of the LB-2 coating, coating failures were not observed in 10 hours at 2000°F on any specimens deflected to angles of 2 and 5 degrees. Failure of the LB-2 coating was general and could not be attributed to the localized prestrained condition. Excepting the Chromalloy coated specimen, failures occurred in less than 10 hours at 2000°F on all specimens prestrained to a bend angle of 10 degrees.

Cyclic oxidation at 2000°F was then continued until coating failure or to a maximum of 72 hours on all specimens which had not failed within the 10 hour exposure. These specimens are shown in Figure 18 with the failure times noted in parenthesis if less than 10 hours. These data are tabulated in Table 6. In general, coating failures which occurred after 20 hours exposure at 2000°F on specimens deflected 2 and 5 degrees, could not be directly attributed to the room temperature prestrain. It is significant to point out, however, base metal plastic deformation to the extent produced by a 5 degree bend deflection did not reduce the 2000°F protective life of the majority of the coating-D-14 alloy systems tested.

A second group of coated specimens prestrained at room temperature were exposed in air for 2 hours at 2600°F. The LB-2 coated specimens failed and melted in less than 2 hours at 2600°F, as was the case with the LB-2 coated oxidation coupons. Only the GT&E and TRW coated specimens did not show coating failures after 2 hours at 2600°F. Again the deflection of 2 and 5 degrees had no apparent influence on the location of coating failures.

Cyclic oxidation at 2600°F was then continued on those specimens which had not failed after the 2 hour exposure, and was terminated at 30 hours. Figure 19 is a photograph of these specimens. All specimens except those of GT&E and TRW failed in less than 10 hours. These data are listed in Table 6.

2000°F


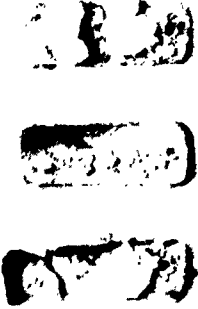
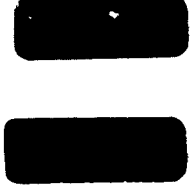

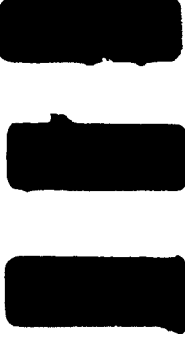
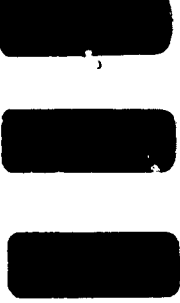
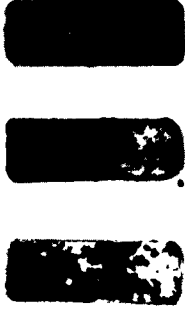
 <p>72 72 11 Hours 5 10 2 Degrees Bend</p> <p>Chromalloy</p>	 <p>10(6) 10(7) 10(3) Hours 5 5 10 2 Degrees Bend</p> <p>McDonnell</p>	 <p>72 14 10(3) Hours 5 10 2 Degrees Bend</p> <p>Pfaudler</p>	 <p>72 10(7) 10(8) Hours 5 10 2 Degrees Bend</p> <p>Pratt &amp; Whitney</p>
 <p>31 52 10(6) Hours 5 10 2 Degrees Bend</p> <p>GT&amp;E</p>	 <p>72 72 10(1) Hours 5 10 2 Degrees Bend</p> <p>TRW</p>	 <p>72 14 10(1) Hours 5 10 2 Degrees Bend</p> <p>Vought</p>	

Figure 18 Coated D-14 Alloy Bend Specimens Cyclic Oxidation Tested to Failure or to a Maximum of 72 Hours at 2000°F after Room Temperature Prestraining to Bend Angles of 2, 5, and 10 degrees. Failure Time Indicated in Parenthesis if Less Than 10 Hours





TABLE 6

Protective Life Of Various Coatings On D-14 Alloy  
At 2000 And 2600°F After Prestraining To  
Bend Angles of 2, 5 and 10 Degrees

Coating Organization	Protective Life - Hours(1)					
	2000°F		2600°F		2600°F	
	2°	5°	10°	2°	5°	10°
Chromalloy	72	72	11	11	10	1
McDonnell	6	7	3	2	2	2
Pfandler	72	14	3	5	5	2
Pratt & Whitney	72	7	8	5	2	5
GT&E	31	52	6	10	30	26
TRW	72	72	1	30	30	22
Vought	72	14	1	2	5	2

(1) Oxidation exposure terminated after 72 hours  
at 2000°F and 30 hours at 2600°F

2600°F

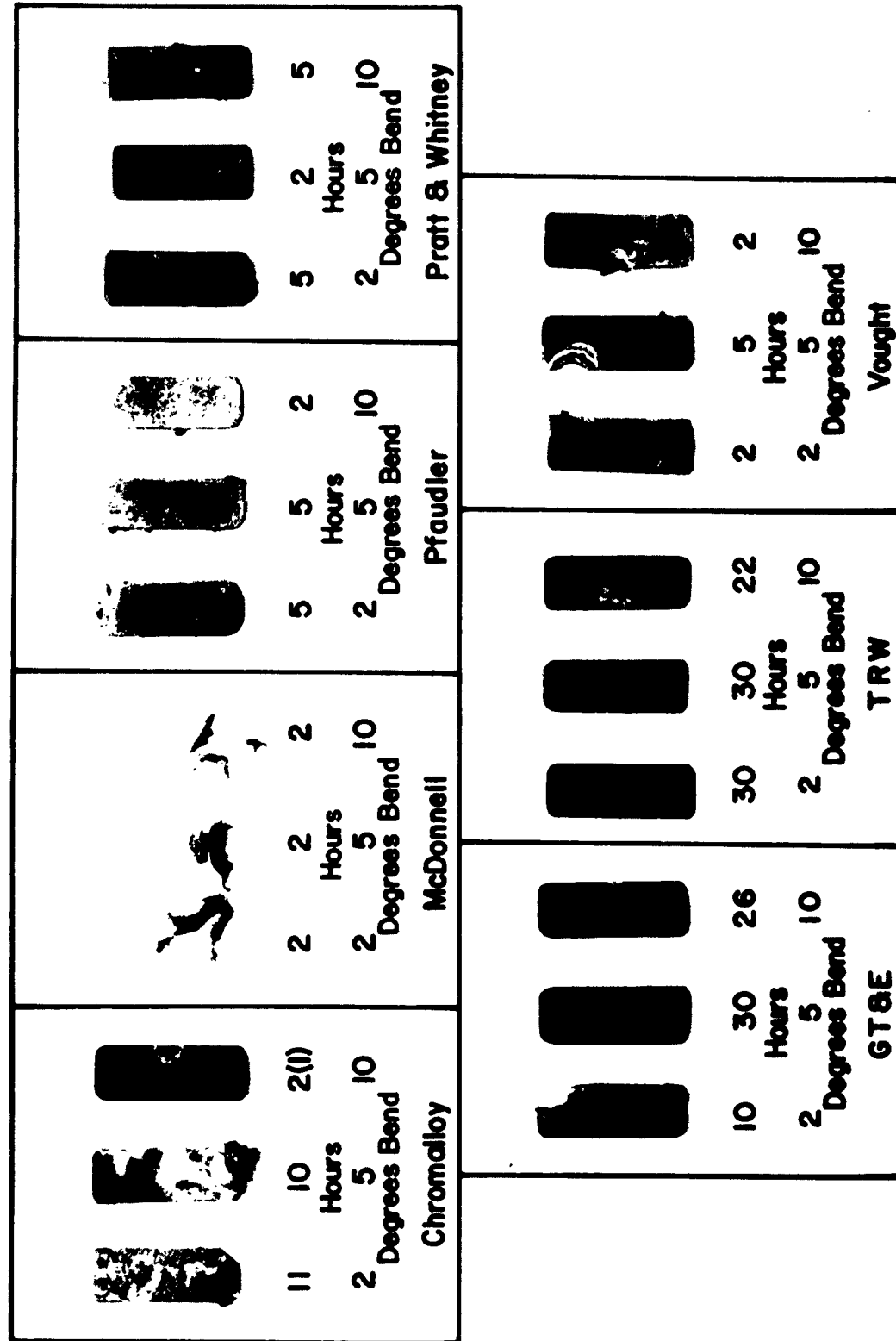


Figure 19 Coated D-14 Alloy Bend Specimens Cyclic Oxidation Tested to Failure or to a Maximum of 30 Hours at 2600°F after Room Temperature Prestraining to Bend Angles of 2,5, and 10 Degrees. Failure time Indicated in Parenthesis if Less than 2 Hours. 1X



In all cases, failures which occurred on specimens prestrained 10 degrees were initiated in the deformation region. However, it can be noted that the GT&E and the TRW coatings on these specimens were protective for longer times at 2600°F than at 2000°F, indicating clearly that self-healing of coating flaws did occur at the higher temperature.

The relative deformation tolerances of the seven coatings were generally in the same order as that indicated by the comparative protective characteristics established by the cyclic oxidation tests. This might be anticipated since the cyclic oxidation test reflects both the resistance of the coating to progressive oxidation and the ability of the coating to withstand or deform under thermally induced stresses.

#### 4.4 Selection of Coating-Alloy Systems for the Design Data Study

The results of the five screening tests on the seven oxidation protective coatings applied to D-14 alloy were carefully reviewed and analyzed in a joint meeting between TRW and Bureau of Naval Weapons personnel. Based generally on the overall protective characteristics of the seven coatings over the temperature range 1600-2600°F, a tentative selection was made of the Pfudler, TRW and Vought coatings for evaluation in the design data study.

As mentioned previously, selection of the sheet alloy for the design data portion of the program was based primarily on availability and current interest to the Refractory Metal Sheet Rolling Panel, MAB. Three alloys were given serious consideration: B-66 (Cb-5V-5Mo), Fansteel 85 (Cb-28Ta-10W-1Zr) and X-110 (Cb-10W-1Zr). The delivery schedule for 30 mil FS-85 alloy coincided most favorably with the time schedule of this program.

In view of the considerable difference in composition of the D-14 and FS-85 alloys and the expense of preparing a large number of test specimens for the mechanical property investigation, a brief series of preliminary evaluation tests were conducted to evaluate the three candidate coatings on FS-85 alloy. Experience has shown that regardless of the alloy being evaluated the order of protective performance of various coatings is generally unchanged. However, the ultimate protective characteristics of all coatings are enhanced by their application on the relatively more oxidation resistant columbium alloys. These preliminary tests of the three coatings on FS-85 alloy were performed to investigate the presence of any anomaly in the protective nature of the three coatings on this particular alloy. The four evaluation tests employed were briefly as follows:

1. Metallography
2. Room temperature bend ductility tests on uncoated and coated FS-85 alloy sheet
3. Cyclic oxidation tests at 2000 and 2600°F
4. Room temperature prestrain to bend angles of 2 and 5 degrees followed by cyclic oxidation at 2000 and 2600°F

#### 4.5 Preliminary Evaluation Tests on Coated FS-85 Alloy

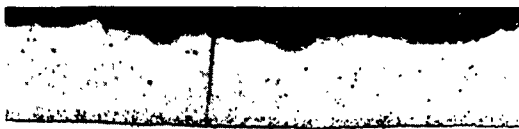
Twelve FS-85 alloy specimens were coated by Pfaudler, TRW and Vought and subjected to the four evaluation tests described above. The specimens were prepared utilizing the same procedure discussed in Section 4.2 with regard to the D-14 alloy. The test procedures employed in these tests were identical with those utilized in the screening evaluation of the seven coatings on D-14 alloy (Section 4.3).

##### 4.5.1 Metallography of Coated FS-85 Alloy

One specimen representing each of the three coatings was sectioned and metallographically prepared. Figure 20 shows photomicrographs of the three coatings in the as coated condition. The coating thicknesses were as follows: Pfaudler (2.0-2.5 mils), TRW (3.0-3.5 mils) and Vought (4.0-4.5 mils). The Pfaudler coating on FS-85 alloy was slightly thinner than that previously deposited on D-14 alloy, whereas both the TRW and Vought coatings were slightly thicker. A comparison of these photomicrographs with those of Figures 1 and 2 for D-14 alloy indicate differences are exhibited for the microstructures of the three coatings formed on these two alloys.

##### 4.5.2 Cyclic Oxidation Tests

Cyclic oxidation tests were conducted to failure or to a maximum of 150 hours at 2000 and 2600°F, on three specimens of each of the three coatings. Figure 21 shows a photograph of three specimens of each coating after cyclic oxidation for a maximum of 150 hours at 2000°F. A spot failure occurred on one Pfaudler coated specimen after 45 hours at 2000°F, however, all other specimens were protected for the 150 hour duration of the test. These protective lives (in excess of 150 hours) at 2000°F



Pfaudler

2.25 Mils



TRW

3.25 Mils



Vought

4.25 Mils

Figure 20 Photomicrographs of Coatings on FS-85 Alloy  
in the As Coated Condition, Including  
Average Coating Thickness Measurements 250X

2000°F










Pfaudler			T R W			Vought		
								
45	150	150	150	150	150	150	150	150
Hours			Hours			Hours		

Figure 21 Coated FS-85 Alloy Specimens Cyclic Oxidation Tested to Failure or to a Maximum of 150 Hours at 2000°F LX



represent equivalent protective characteristics for the TRW and Vought coatings on both D-14 and FS-85 alloys, and an increase in the protective life of the Pfaudler coating on FS-85 relative to comparable tests on D-14 alloy (5, 66 and 69 hours for the Pfaudler coating on D-14 alloy).

Figure 22 shows the coated FS-85 specimens after cyclic oxidation testing at 2600°F. The Vought coating failed in less than one hour at 2600°F resulting in the formation of a molten oxide slag which destroyed the entire coupon. This represents a considerable decrease in protective performance of the Vought coating as compared to the same coating applied to D-14 alloy where the failure times were 6, 13 and 13 hours. The Pfaudler coating on FS-85 alloy failed at 3, 6 and 9 hours at 2600°F as compared to lives of 9, 11 and 11 hours on D-14 alloy. Only the TRW coating showed greater protective lives at 2600°F on FS-85 alloy as compared to D-14 alloy, with three Cr-Ti-Si coated FS-85 alloy specimens showing no failures after 72 hours of cyclic oxidation at 2600°F.

#### 4.5.3 Room Temperature Bend Ductility Tests

Bend tests were conducted on uncoated and as coated FS-85 alloy specimens at room temperature. The Instron machine cross-head speed was 10 inches per minute in accordance with MAB test specifications. Both Vought coated FS-85 alloy specimens were brittle as coated, which was also found in the case of the D-14 alloy. One TRW specimen gave a 90° bend without cracking while the second specimen failed with a brittle fracture. Both Pfaudler coated specimens cracked after approximately a 70° bend angle. The bend transition temperature for both the Pfaudler and TRW coated FS-85 alloy was near room temperature for these test conditions, whereas, both were well below room temperature in the case of D-14 alloy tested in the initial screening evaluation. Bend tests on uncoated FS-85 alloy sheet at room temperature gave 90° bend angles in both the longitudinal and transverse directions, however, surface cracks were evident on those specimens bend tested in the longitudinal direction. Obviously, the bend transition temperature of the uncoated FS-85 was higher than that of the D-14 alloy tested in this program. The FS-85 alloy was, therefore, considerably more sensitive to the coating heat treatments and brittle coating surface layers. These cracks in the uncoated sheet were quite significant and will be discussed in the following section.

#### 4.5.4 Room Temperature Prestrain + Cyclic Oxidation Tests

FS-85 specimens representing each of the three coatings

2600°F










Pfaudler			T R W			Vought		
								
3	6	9	72	72	72	1	1	2
Hours			Hours			Hours		

Figure 22 Coated FS-85 Alloy Specimens Cyclic Oxidation Tested to Failure or to a Maximum of 72 Hours at 2600°F LX





were prestrained at room temperature to bend angles of 2 and 5 degrees and subsequently cyclic oxidation tested for 10 hours at 2000°F and for 2 hours at 2600°F. No coating failures were observed after 10 hours of oxidation at 2000°F. Oxidation tests were continued on these specimens to a maximum of 100 hours at 2000°F and the specimens are shown in Figure 23. Only the Pfadler coated specimen prestrained to a bend angle of 5° failed in less than 100 hours, showing a localized failure in the bend area after 30 hours at 2000°F. As was previously shown in the case of coated D-14 alloy, prestraining at room temperature to bend angles of 2 and 5° had no definite detrimental effect upon the protective life of the three coatings at 2000°F.

A similar set of prestrained FS-85 alloy specimens were oxidation tested for two hours at 2600°F. Again both of the Vought coated specimens failed and melted in less than one hour at 2600°F. The oxidation tests were continued and failures were observed after 4 and 2 hours on the 2 and 5° prestrained Pfadler coated specimens, respectively. No coating failures were observed on the prestrained TRW coated FS-85 specimens after 48 hours at 2600°F and the tests were terminated. These specimens are shown in Figure 24.

In general then, all three coatings showed equivalent or better protection at 2000°F on FS-85 as compared to their performance in similar tests on D-14 alloy. At 2600°F, however, the TRW coating applied to the FS-85 alloy was considerably more protective than in the case of D-14 alloy, whereas, the Pfadler coating was somewhat less protective on FS-85 alloy and the Vought coating afforded FS-85 alloy virtually no protection at all. Room temperature bend tests indicated that all three coating treatments raised the bend transition temperature of the FS-85 alloy near or above room temperature.

These data were again reviewed in a joint meeting between TRW and Bureau of Weapons personnel. It was mutually agreed that in view of the severe high temperature (2600°F) limitation of the Vought coating on FS-85 alloy the design data study would be conducted utilizing only the Pfadler and TRW coatings.

As a result of the unfavorable bend test data generated on the coated and uncoated FS-85 alloy sheet in this brief evaluation, the chemical analysis of this heat of material was re-investigated by the Fansteel Metallurgical Corporation. The carbon content was found to be 250 ppm, as compared to 50 ppm previously reported upon receipt of the entire lot of sheet.

2000°F

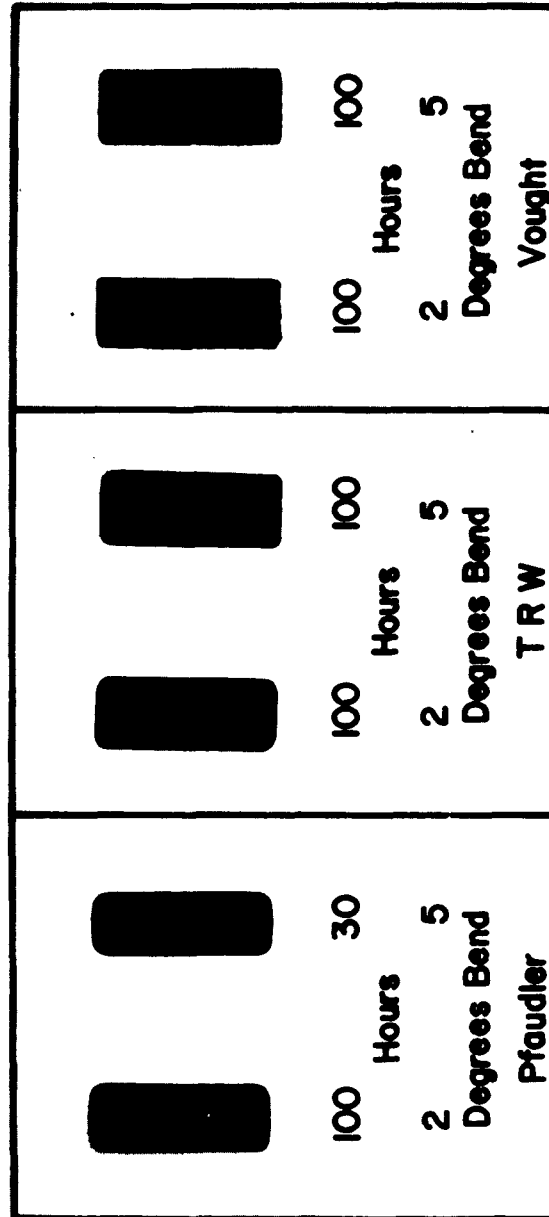


Figure 23 Coated FS-85 Alloy Specimens Prestained at Room Temperature to Bend Angles of 2 and 5° Followed by Oxidation Testing to Failure or to a Maximum of 100 Hours at 2000°F 1X

2600°F




Pfaudler			TRW			Vought		
	4	Hours		48	Hours		1	Hours
	2	Degrees Bend		2	Degrees Bend		2	Degrees Bend
							5	Degrees Bend
								Vought

Figure 24 Coated FS-85 Alloy Specimens Prestained at Room Temperature to Bend Angles of 2 and 5° Followed by Oxidation Testing to Failure or to a Maximum of 48 Hours at 2600°F LX

A similar chemical analysis at TRW indicated a carbon content of approximately 160 ppm.

The current target properties for columbium base materials established by the Materials Advisory Board Refractory Metal Sheet Rolling Panel include a 15% elongation requirement for room temperature tensile tests. In order to determine any detrimental influence of the high carbon content on the tensile ductility, room temperature tensile tests were conducted on the FS-85 alloy sheet in the following three conditions: as received (stress relieved), recrystallized in vacuum for one hour at 2300°F and recrystallized and Cr-Ti-Si coated. These data are reported in Table 7.

In the wrought condition the FS-85 alloy sheet did not meet the target specification of the MAB panel. However, after both the recrystallization and coating heat treatments elongation values were obtained which were in excess of the 15% minimum, along with approximately a 30% reduction in the corresponding ultimate and yield strengths.

The Cr-Ti-Si coating treatment involved Cr-Ti coating for 8 hours at 2300°F followed by siliconizing for 4 hours at 2100°F, this treatment obviously resulted in full recrystallization of the FS-85 alloy sheet. Figure 25 shows photomicrographs comparing the structural changes resulting from these heat treatments. The somewhat less elongation realized with the coated sheet as compared to the recrystallized (uncoated) material was attributed to two factors: 1) additional grain growth resulting from the 8 hour treatment at 2300°F, and 2) the notch effect and complex stress state imposed by the relatively brittle coating on the specimen surface.

In view of the time schedule for this program and the favorable ductility change resulting from recrystallization of the FS-85 alloy, the decision was made to continue the design data study utilizing this heat of FS-85 material.

##### 5. DESIGN DATA STUDY

The sensitivity of columbium alloys and other body centered cubic metals to mechanical property alterations resulting from thermal treatment, interstitial contamination, microstructural changes, etc., is well recognized. These factors complicate considerably the utilization of refractory metal metallurgical property data in design considerations. The necessity of pro-



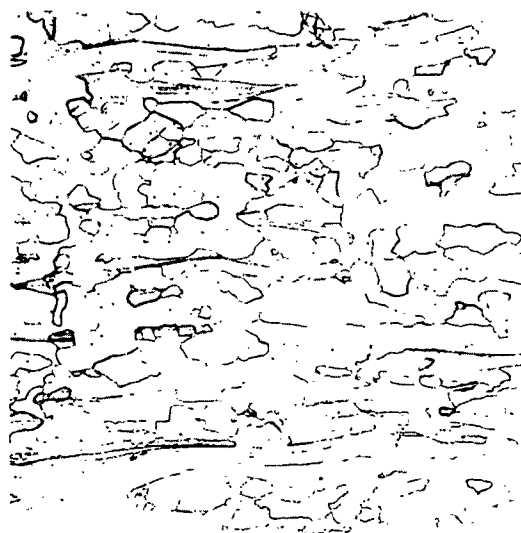
TABLE 7

Room Temperature Tensile Properties  
For 30 Mil FS-85 Alloy Sheet

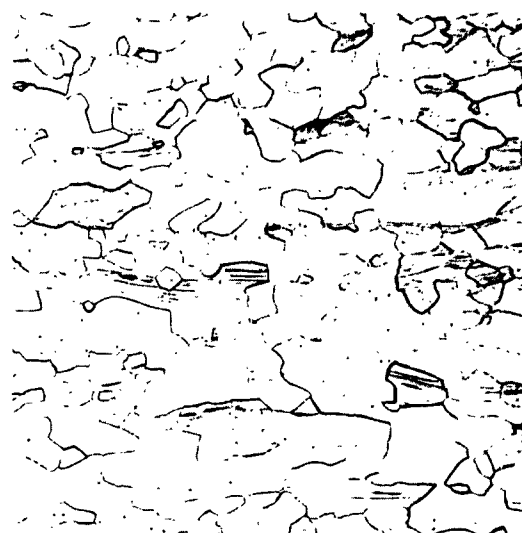
<u>Condition</u>	<u>UTS-PSI<sup>(1)</sup></u>	<u>0.2% Offset Y.S.-PSI</u>	<u>% Elongation in 1" Gage</u>	<u>% R.A.</u>
As Received (stress relieved)	119,400	109,500	10.5	42.9
	120,400	106,800	11.0	47.6
	120,500	104,800	11.0	42.8
Recrystallized 1 hour-2300° F vacuum	86,900	69,500	21.5	46.7
	85,300	69,200	22.0	54.0
	85,600	69,400	23.0	54.7
Recrystallized and Cr-Ti-Si Coated <sup>(2)</sup>	84,200	68,400	18.0	32.8
	83,800	68,600	17.5	32.5
	80,400	65,700	16.0	25.1

(1) Strain rate - 0.020 in/in/min.

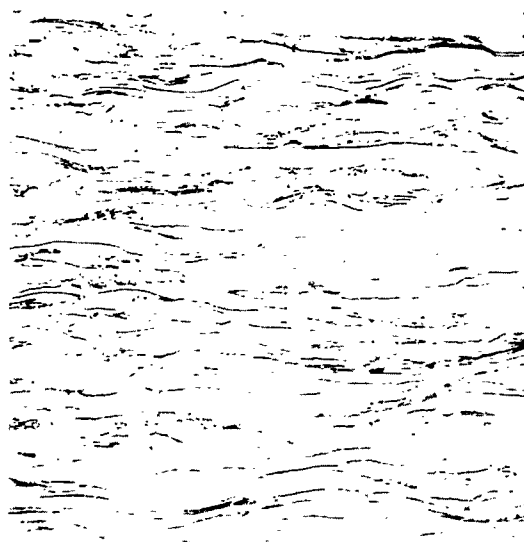
(2) Strength and ductility calculations based on original  
uncoated gage dimensions



1 Hour - 2300°F



8 Hours - 2300°F  
4 Hours - 2100°F



As Received

Figure 25 FS-85 Alloy as Received, after Recrystallization  
for 1 Hour at 2300°F and as Cr-Ti-Si Coated 250X



testing these metals against atmospheric contamination and erosion during service requires the use of protective coatings, which adds the further complication of requiring an evaluation of the composite coating-base metal mechanical properties. The micro-structural changes resulting from the coating application treatment, the diffusion of coating elements into the substrate, reduction of the base metal load bearing cross section, the notch effect created by stress induced cracks in the brittle surface layer and many other factors related to the protective surface alloy layers greatly influence the metallurgical design properties of the coating base metal systems. The evaluation tests involved in this study comprise only a portion of the investigations required to generate useful and comprehensive design data of composite coating - columbium alloy systems.

Section 4 presented a thorough discussion of the coating screening tests conducted prior to the design data study. Protective coatings applied by Pfaudler and TRW were selected for the mechanical properties evaluation based on the results of screening studies of seven oxidation protective coatings applied on D-14 and FS-85 alloys. FS-85 alloy was selected as the columbium alloy sheet material for this investigation, based both on reported properties and on material availability.

Section 5 involves a thorough presentation of the results of the design data study in tabular, graphical and pictorial form for analysis by the reader. Observations of coating performances and generalized property comparisons are presented where applicable, however, specific statements regarding relative performances of the two coatings throughout the various tests have been omitted in view of the presence of the TRW coating in the testing program.

#### 5.1 Testing Program

Five evaluation tests were designed to investigate both the general protective characteristics of the two coatings and their influence on the mechanical properties of FS-85 alloy. These tests were as follows:

- 1) Cyclic oxidation of four specimens of each coating in air at 1600, 1800, 2000, 2300, 2500 and 2600°F, to failure or to a maximum of 150 hours
- 2) Thermal shock-erosion-oxidation tests for 100, 250 and 500 cycles from 2600-250°F followed by post-oxidation for 2 hours at 1600°F to accentuate coating failures

- 3) Tensile tests on uncoated (vacuum) and coated (air) FS-85 alloy sheet at RT, 400, 800, 1200, 1600, 1800, 2000, 2300, 2500 and 2600°F
- 4) Prestraining of coated tensile type specimens in the base metal elastic and plastic deformation ranges at RT, 400, 800, 1200, 1600, 2000, 2300 and 2600°F followed by cyclic oxidation testing for 2 hours at 1600 and 2600°F to determine the strain tolerance of the coatings at various temperature levels
- 5) Stress rupture tests of coated FS-85 alloy at five (5) stress levels at 1600, 1800, 2000, 2300, 2500 and 2600°F for a maximum of 150 hours of exposure.

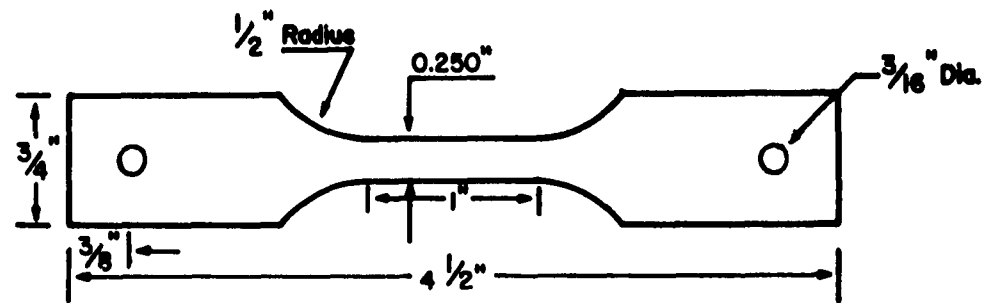
#### 5.2 Specimen Preparation

Preparation of the FS-85 alloy evaluation specimens for coating involved the following procedure:

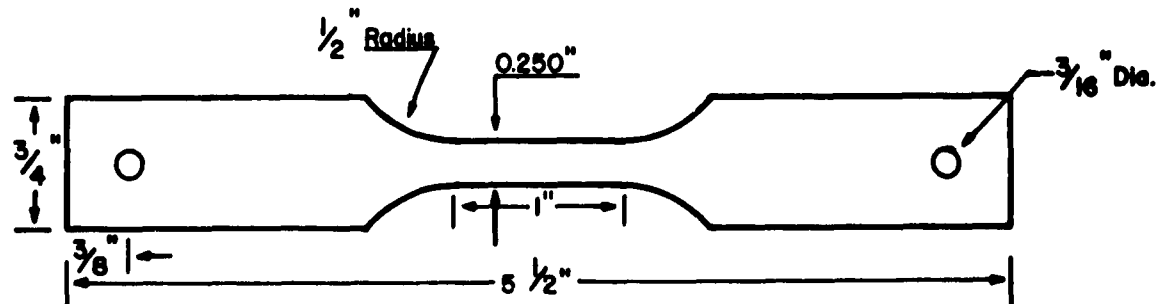
- 1) Machined various specimen configurations shown in Figure 26
- 2) Produced uniform radii on all specimen edges and corners by an abrasive tumbling technique
- 3) Degreased in trichloroethylene
- 4) Lightly etched in an  $\text{HF-HNO}_3\text{-H}_2\text{SO}_4$  aqueous solution and rinsed in water
- 5) Recrystallized in vacuum for 1 hour at 2300°F.

As reported in Section 4.5, recrystallization of the FS-85 alloy rendered this material more ductile than the wrought condition at low temperatures, and was necessary in order to meet a 15% room temperature elongation requirement established by the Materials Advisory Board, Refractory Metal Sheet Rolling Panel.

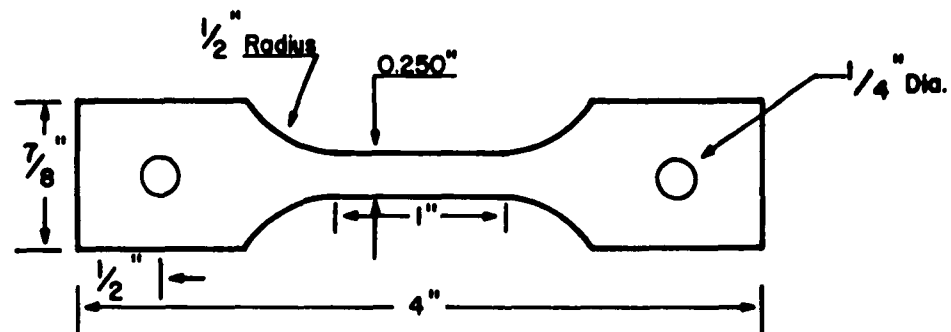




A · TENSILE SPECIMEN - RT to 2300°F



B · TENSILE SPECIMEN - 2500 and 2600°F



C · STRESS RUPTURE SPECIMEN

Figure 26 Drawings of Tensile and Stress Rupture Specimen Configurations Used in the Design Data Study

The Pfaudler Company reported the following processing information with regard to coating this lot of evaluation specimens. The coating was applied by pack cementation methods utilizing dual cycles of 7 hours at 2050°F. A barrier coating inner layer was first deposited and then overlaid with a modified silicide outer layer. The specimens were processed in five (5) separate double cycle lots. Information was provided with regard to specimen location within the retort and this identification was retained throughout the testing program. The only additional specimen preparation performed by Pfaudler prior to coating involved washing, lightly etching, rewashing and rinsing in acetone. Figure 27 is a photograph showing a layout of the group of specimens coated by Pfaudler. All specimens were inspected under 30X magnification and individually catalogued prior to testing. Five (5) tensile type specimens of the two hundred and twelve (212) specimens coated by Pfaudler were defective. Spalling of the coating was observed on these specimens in the as received condition.

The TRW Cr-Ti-Si alloy coating was applied on the FS-85 alloy specimens utilizing the following procedure:

- 1) Degreased the vacuum recrystallized specimens in trichloroethylene and washed in acetone
- 2) Deposited the Cr-Ti alloy coating from a 50Cr-50Ti alloy pack in 8 hours at 2300°F using KF pack activation
- 3) Siliconized the Cr-Ti alloy layer for 4 hours at 2000°F in a KF activated metallic silicon pack.

All specimens were processed through the double coating cycle in one (1) lot. Figure 28 is a photograph showing a layout of these specimens. There were no visibly evident (30X) coating defects on any of the two hundred and twelve (212) Cr-Ti-Si coated specimens.

Random specimens of each coating were sectioned and metallographically prepared. Figure 29 shows photomicrographs of both the Pfaudler and TRW (Cr-Ti-Si) coatings on FS-85 alloy. The Pfaudler coating ranged in thickness from 1.5-3.2 mils (Figure 29 shows the variation in coating thickness) and the TRW Cr-Ti-Si coating from 2.5-3.0 mils. As will be evident in the subsequent sections, the non-uniformity in thickness of the Pfaudler coating introduced a factor of inconsistency in the test results involving the protective characteristics of the coating.

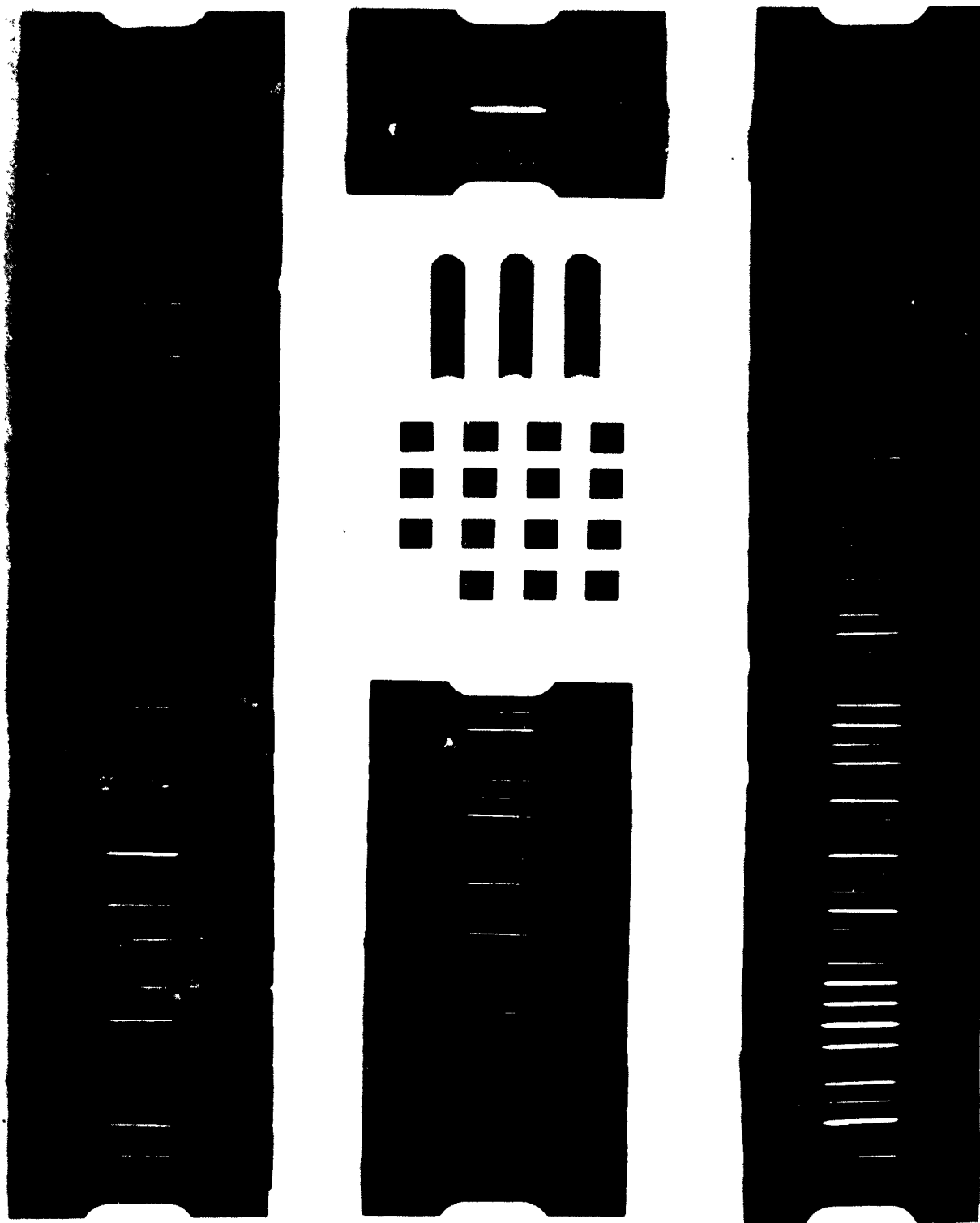


Figure 27 Photograph of FS-85 Alloy Evaluation  
Specimens Coated by Pfaudler Company  
Approximately 1/3X

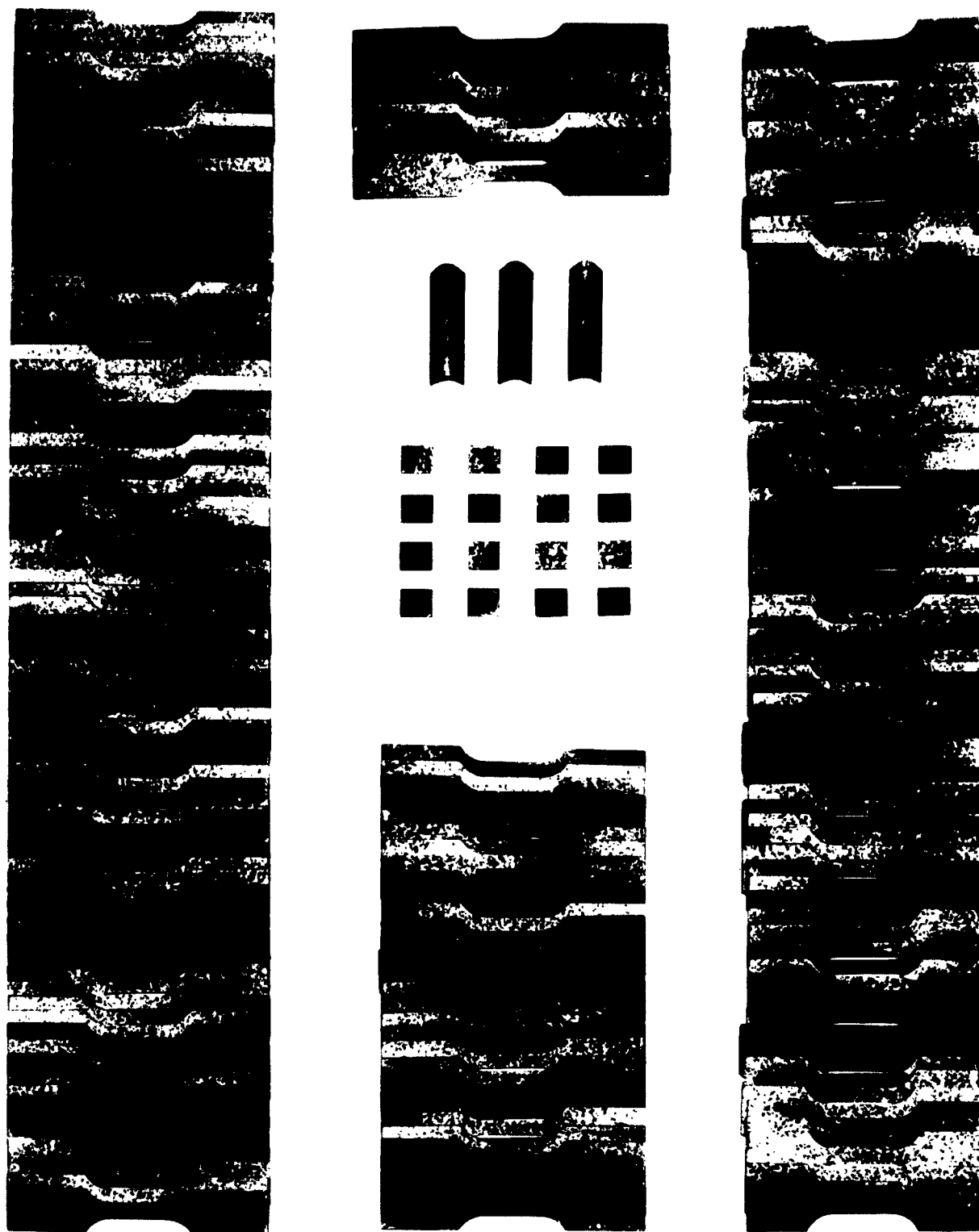
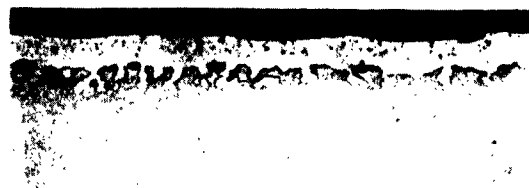


Figure 28 Photographs of FS-85 Alloy Evaluation  
Specimens Cr-Ti-Si Coated by TRW  
Approximately 1/3X



Cr-Ti

Cycle #1

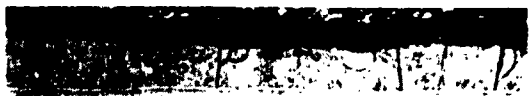
0.5 Mil

Cr-Ti-Si

Cycle #2

2.7 Mil

TRW Coating



Thin Area

1.5 Mil

Thickest Area

3.2 Mil

Pfaudler Coating

Figure 29 Photographs of Pfaudler and TRW Coatings on FS-85 Alloy, Showing the Coatings Produced in Both Cycles of the TRW Coating Process and Variation of Coating Thickness in the Pfaudler Coating 250X

### 5.3 Cyclic Oxidation

Cyclic oxidation tests were performed on four (4) specimens of each coating at temperatures in the range 1600-2600°F. Cyclic oxidation involved cycling the test specimens from the test temperature to room temperature once each hour for 8 hours, followed by 16 hours of static exposure in each 24 hour period. Failure of the coating was defined as the evidence of columbium oxide erupting from the substrate through the coating. The specimens were placed on alumina pads in platinum boats and exposed in air in global heated box furnaces. All specimens of both coatings were tested simultaneously in the same furnace at each of the individual exposure temperatures. The tests were terminated at all temperatures at a maximum of 150 hours. Table 8 lists the failure times for these tests and photographs of the specimens are presented in Figures 30 and 31.

Both the Pfaudler and TRW coatings were somewhat less protective on this series of specimens than on the FS-85 alloy specimens evaluated in the screening tests and reported in Section 4.5. Both coatings were generally thinner on the latter group of evaluation specimens, which would be expected to reduce the average protective lives.

### 5.4 Thermal Shock-Erosion-Oxidation Tests

The cyclic oxidation test discussed in the previous sections was effectively a thermal shock-oxidation test. The stresses generated at the coating-substrate interface as a result of the intermittent thermal cycling unquestionably produce and propagate cracks through the coatings and reduce their ultimate protective capability as compared to a non-cyclic exposure. However, this type of furnace heating and air cooling might be less severe than the rapid thermal cycle encountered by a coated structure in a re-entry application. The technique utilized here to simulate this type of thermal profile involved heating the specimen surface with an oxidizing oxyacetylene flame to a peak temperature of 2600°F in 20 seconds, followed by cooling in an air blast to approximately 250°F in 20 seconds. The test specimens were curved sheet configurations, positioned such that the flame impinged on the convex surface of the curve radius. Figure 32 is a photograph of the test apparatus and test specimens during operation. A very high initial cooling rate was encountered for approximately the first 1000°F decrease in temperature. Under such conditions of rapid heat transfer and sharp thermal gradients the coating must be subjected to an appreciable thermal stress.



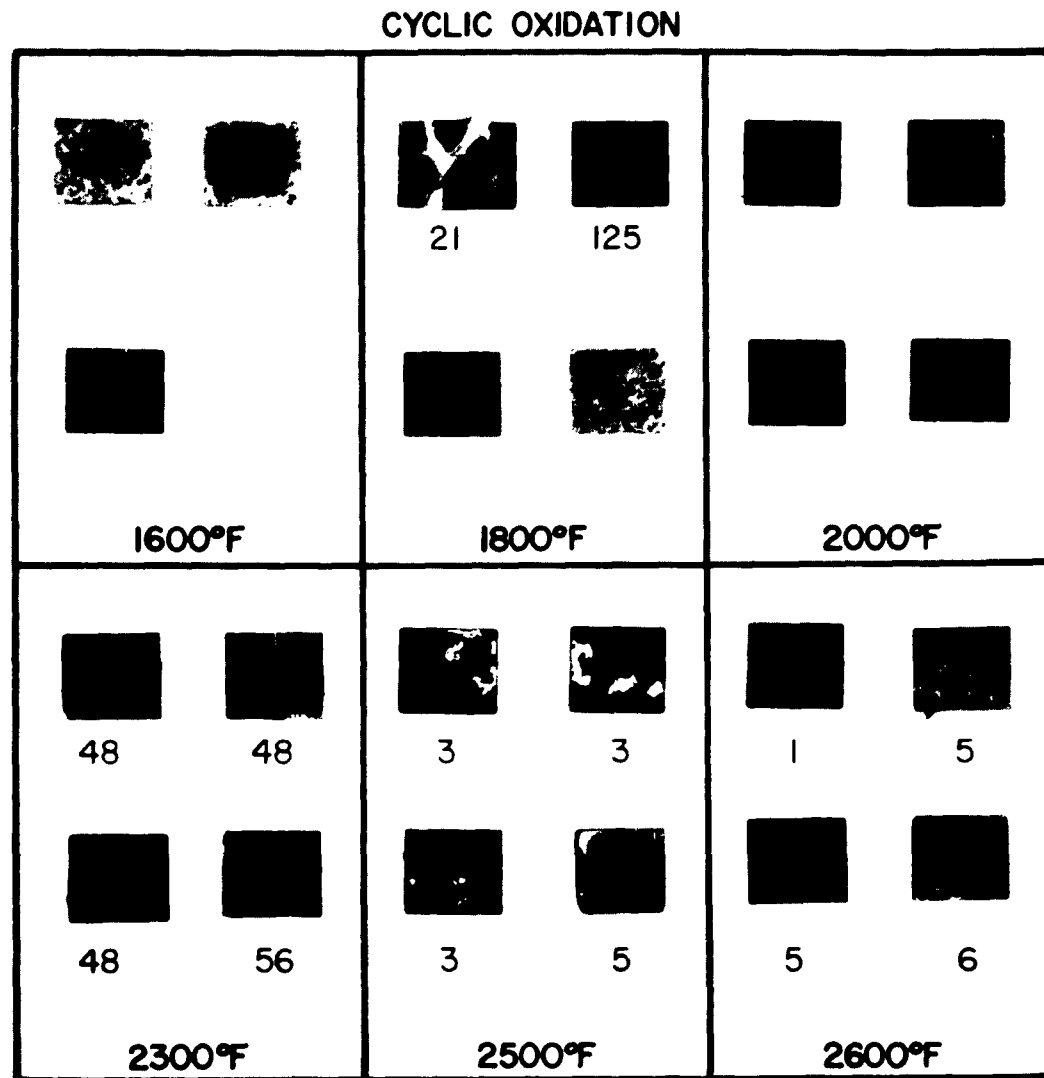
TABLE 8

Protective Lives of Pfaudler and TRW Coatings on  
FS-85 Alloy for Cyclic Oxidation at Temperatures from  
1600 to 2600°F

<u>Oxidation Temperature-°F</u>	<u>Pfaudler</u>	<u>TRW</u>
1600	150, 150, 150(1)	150, 150, 150, 150(2)
1800	21, 125, 150, 150	150, 150, 150, 150
2000	150, 150, 150, 150	150, 150, 150, 150
2300	48, 48, 48, 56	150, 150, 150, 150
2500	3, 3, 3, 5	117, 117, 150, 150
2600	1, 5, 5, 6	45, 51, 51, 51

(1) One specimen not returned, otherwise four specimens tested  
at each temperature

(2) Tests terminated after 150 hours



**Pfautler**

Figure 30 Pfautler Coated FS-85 Alloy Specimens Cyclic Oxidation Tested  
in Air at Temperatures from 1600-2600°F

1X



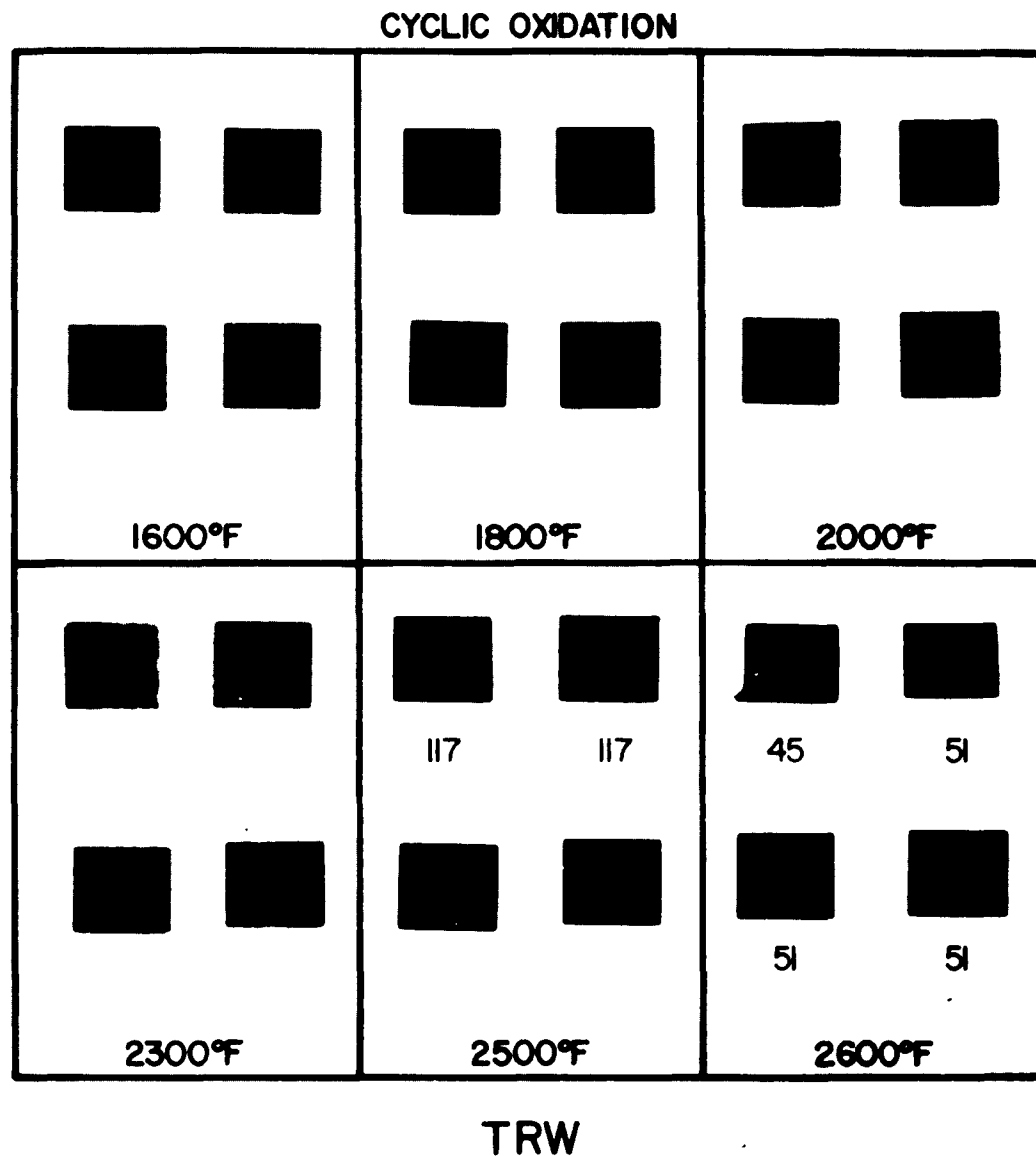


Figure 31 TRW Coated FS-85 Alloy Specimens Cyclic Oxidation Tested in Air at Temperatures from 1600-2600°F

1X

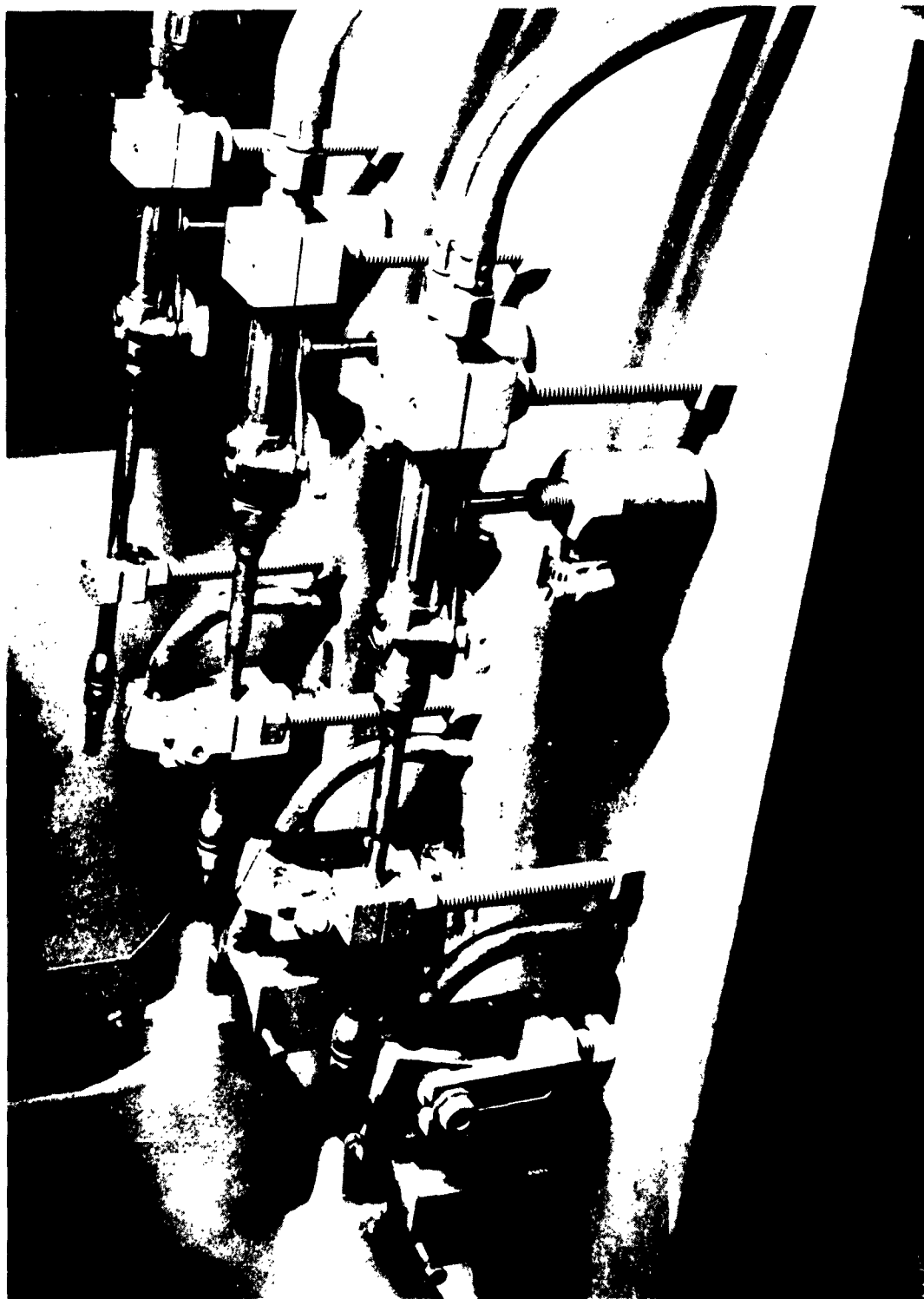


Figure 32 Photograph of Thermal Shock Test Apparatus



Three Pfaudler and three TRW coated FS-85 alloy specimens were subjected to 100, 250 and 500 thermal shock cycles from 2600 to 250°F. These specimens are shown in Figure 33. The Pfaudler coating was observed to spall on the specimens tested for 100 and 500 cycles, however, this spalling was not observed on the specimen cycled 250 times. Craze crack patterns were observed on all specimens in the area of flame impingement, however, columbium oxide growth was not evident at any location.

In order to investigate the existence of thermal shock induced cracks in the coatings all specimens were post-oxidation tested for 2 hours at 1600°F in an air furnace. Figure 34 is a photograph of the specimens after post-oxidation. Gross coating failure occurred in the thermally cycled area on all specimens with the exception of the TRW coated specimen exposed for 100 cycles. Localized coating failure, however, was evident on the concave surface of this specimen.

The specimen surface area adjacent to center of flame impingement represents a coating region thermally cycled from progressively lower peak temperatures. Coating failures in these areas indicate a general sensitivity of both coatings on FS-85 alloy to repeated rapid thermal cycling from a range of temperatures up to 2600°F.

### 5.5 Tensile Properties

The tensile properties of uncoated and Pfaudler and TRW coated FS-85 alloy sheet were determined for the temperature range from room temperature to 2600°F. Prior to tensile testing the uncoated specimens were recrystallized for 1 hour in vacuum at 2300°F. All tests were conducted in an Instron tensile test machine at a constant strain rate of 0.020 inches per inch per minute up to 20% elongation, and then at an increased rate of 0.200 inches per inch per minute until fracture. This strain rate was selected for testing the coated specimens in air, where prolonged heating of the fixturing and heating equipment was undesirable. In order to have comparable data from all tensile tests, this strain rate was also utilized for testing the uncoated sheet in vacuum. Load-extension curves were plotted using one inch gage length test specimens shown as types A and B in Figure 26. The type of specimen heating equipment, which will be discussed subsequently, did not permit the use of a gage extensometer. Where possible, the tensile tests were performed in accordance with the test specifications recommended by the Refractory Metal Sheet Rolling Panel of the Materials Advisory Board for testing uncoated refractory metals in vacuum or in inert atmosphere. Obvious deviations from these specifications were required for testing coated sheet in air.

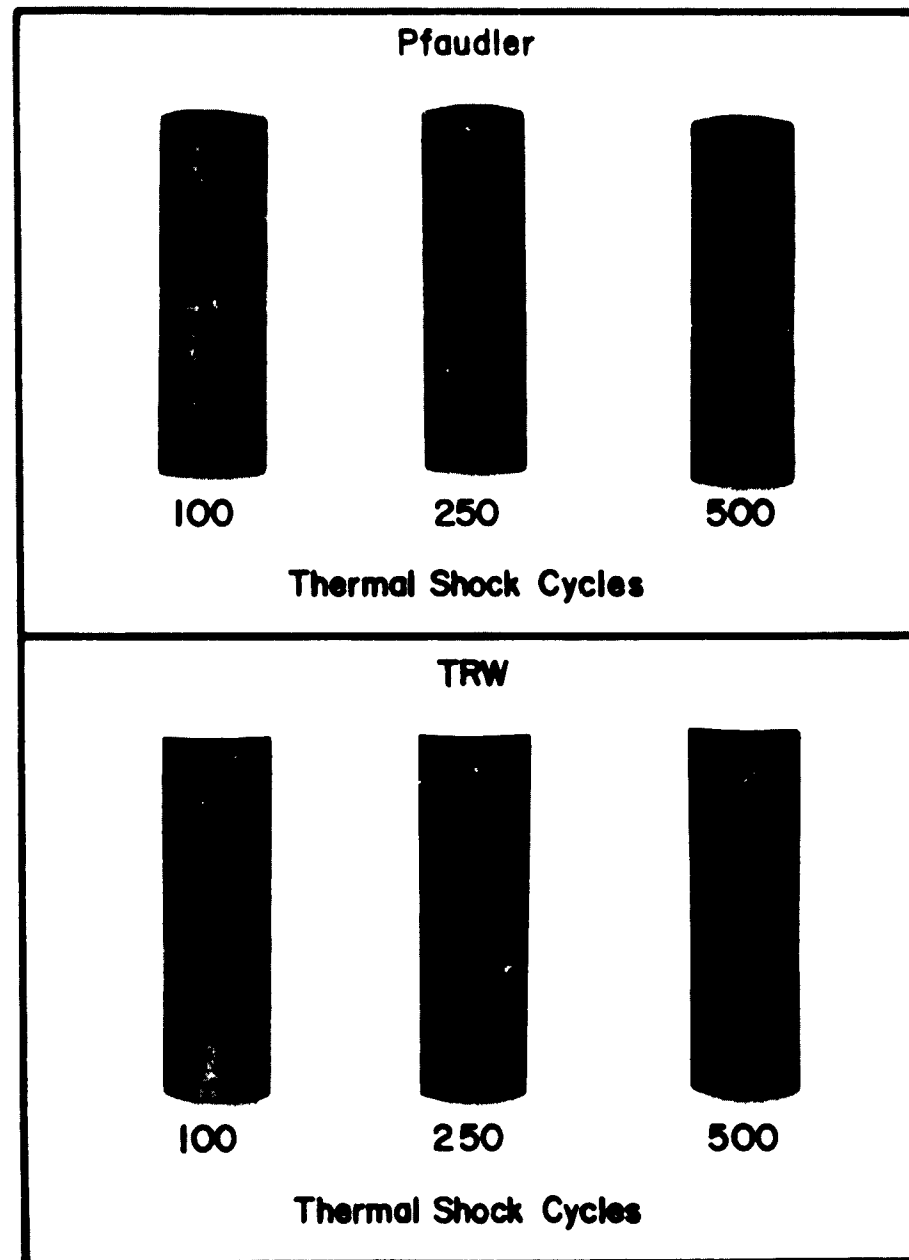


Figure 33 Pfaudler and TRW Coated FS-85 Alloy Specimens Thermal  
Shock Tested by Cycling from 2600 to 250°F 1X

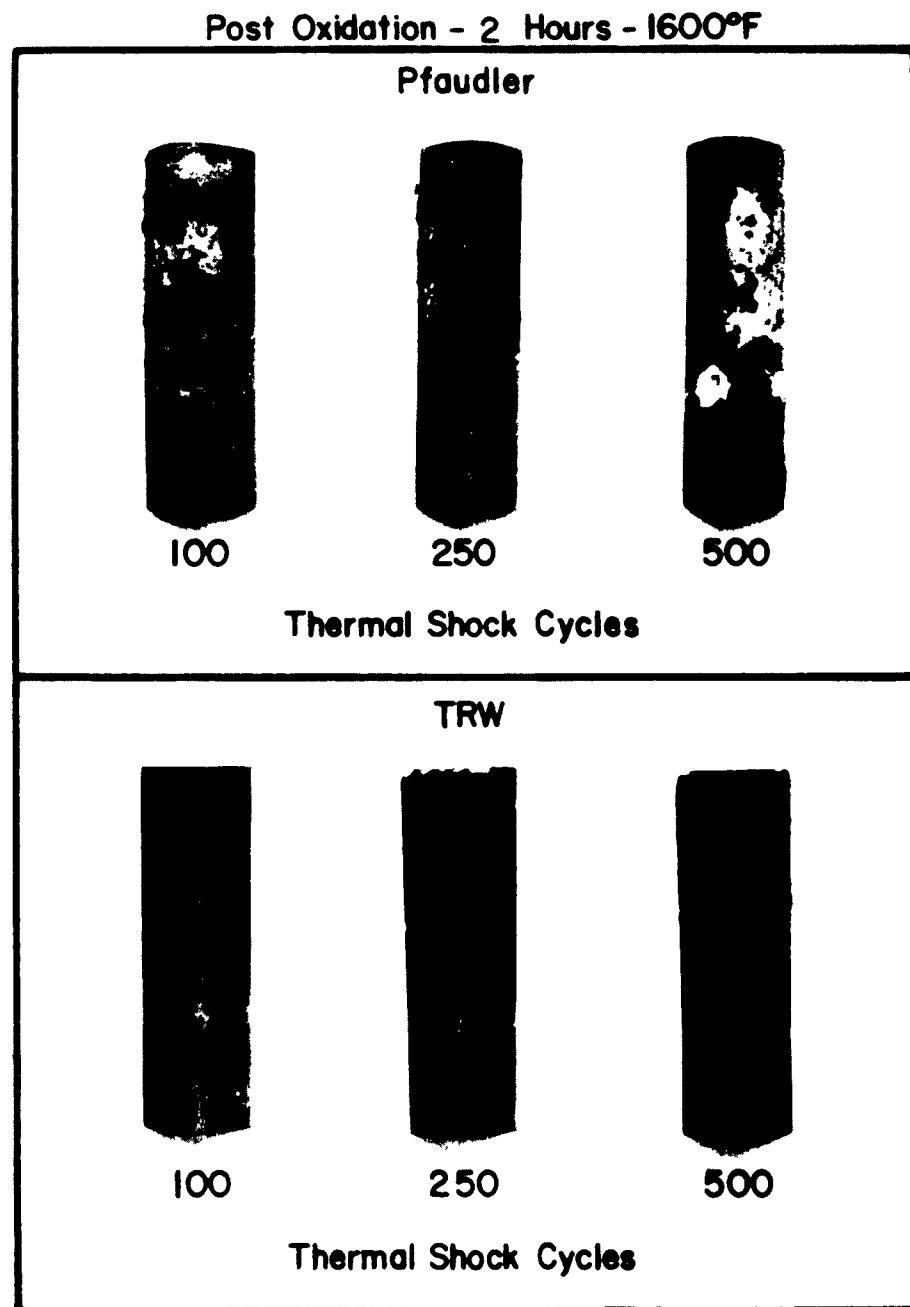


Figure 34 Pfudler and TRW Coated FS-85 Alloy Specimens  
Thermal Shock Tested by Cycling from 2600 to  
250°F, Followed by Post Oxidation for 2 Hours  
at 1600°F

IX

For testing the uncoated specimens at 400 and 800°F a thin ductile aluminum coating was applied in an argon atmosphere at 1400°F prior to testing. The tensile tests were then conducted in air using a resistance wound Marshall tube furnace. At 1200-2600°F the uncoated FS-85 alloy specimens were tensile tested in vacuum, utilizing a resistance heated tantalum element Brew furnace. Platinum-platinum+10% rhodium control thermocouples were spot welded to each specimen. A temperature gradient of 20-30°F existed along the one inch gage section of the specimens heated at the higher temperatures in the Brew furnace.

An infrared radiant heating furnace was utilized for testing the coated FS-85 specimens in air at all temperatures from 400-2600°F. A cross sectional sketch and photograph of this furnace are shown in Figures 35 and 36. The apparatus consisted of a water cooled, rhodium plated cylindrical copper reflector, with eight 500 watt tungsten filament clear quartz lamps providing the radiant energy for heating the centrally located tensile test specimen. In each test the specimen was loaded through Udimet 700 pin type specimen holders which were shielded from direct radiant heating by refractory alumina sleeves over the holders.

Platinum-platinum+10% rhodium control thermocouples were attached to each specimen in the gage section and were shielded from direct outside radiation by a small Cr-Ti-Si coated columbium alloy shield over each thermocouple bead. In all tests the specimens were at heat in less than two (2) minutes and were permitted to soak for a total heating time of ten (10) minutes prior to testing. A temperature gradient of less than 10°F along the one inch gage section was measured at temperatures up to 1800°F, however, at 2000°F and above the gradient increased to 20-30°F.

Tables 9, 10 and 11 list the mechanical property data from this investigation and the properties are plotted as a function of the test temperature in the form of comparative curves in Figures 37 through 40. In general the tensile and yield strengths of the coated FS-85 alloy sheet tested in air were less than those of the uncoated sheet tested in vacuum, however, only in the temperature range from 800 to 1600°F was this differential more than about 5-10%. Original uncoated specimen dimensions were used for the strength calculations with no attempt to compensate for loss of base metal load bearing cross section due to the coating formation. This could account for as much as a 10% apparent reduction in the ultimate tensile and yield strengths.

The strain aging peak exhibited by the uncoated FS-85 alloy at 1200°F was virtually eliminated by application of both the Pfaudler and TRW coatings. Coincident with the strain aging range of the

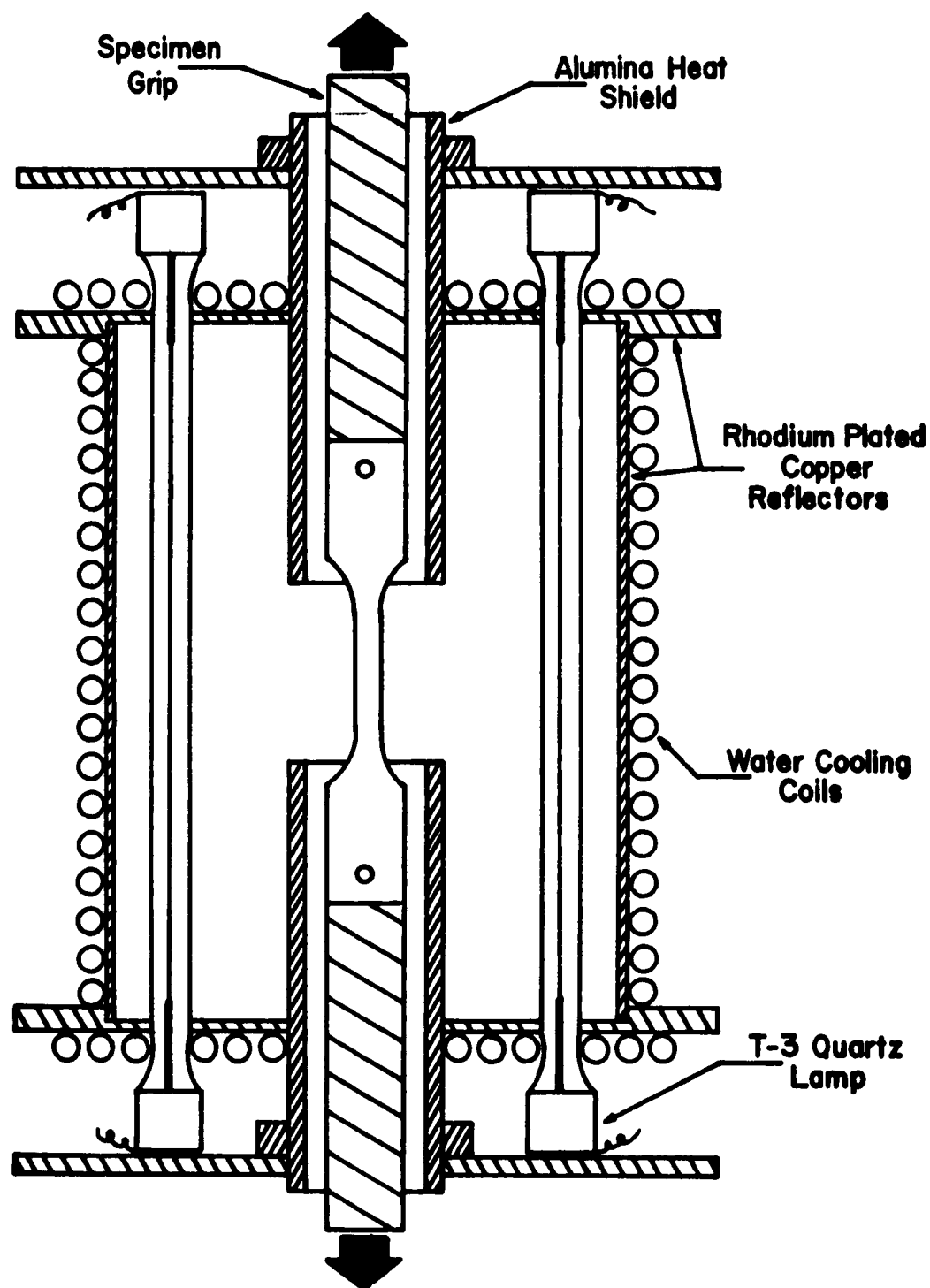


Figure 35 Cross Section of Quartz Lamp Radiant Heated Furnace for Heating Tensile Test Specimens

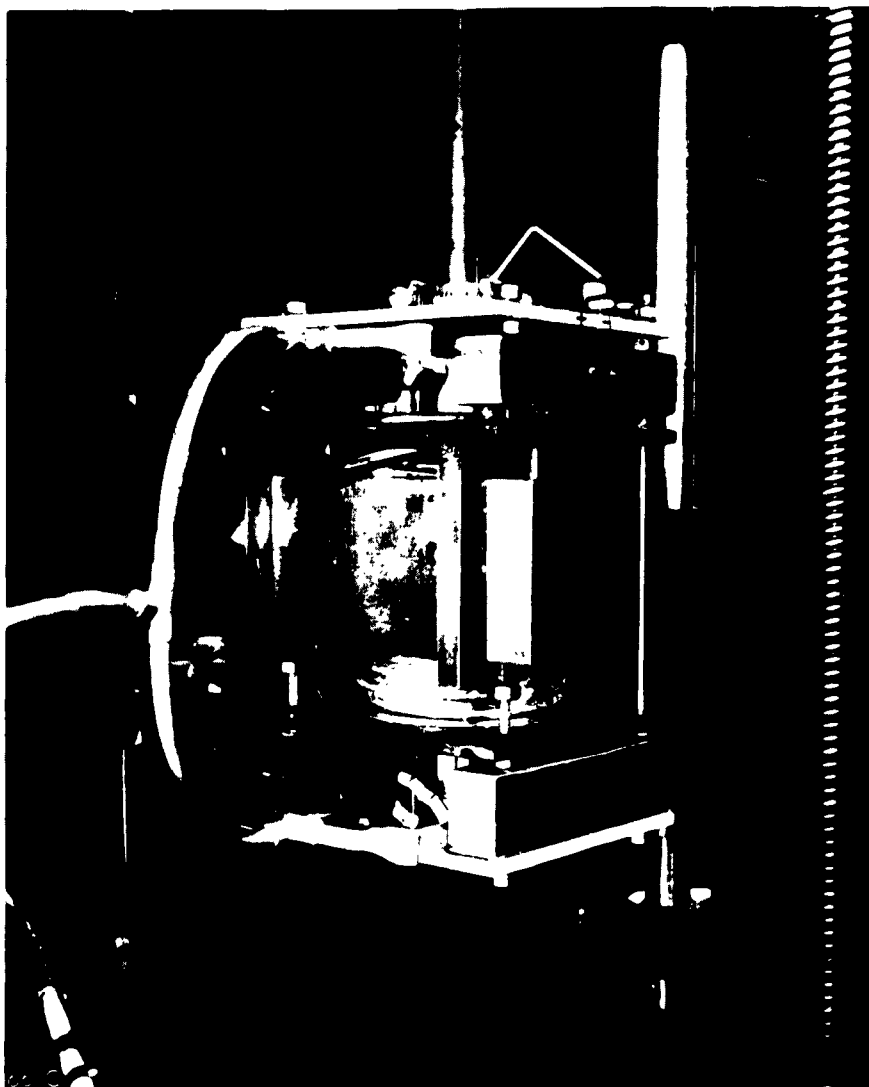


Figure 36 Photograph of Quartz Lamp Radiant Heated Furnace  
Mounted in Instron Tensile Testing Machine





TABLE 9

Tensile Properties of Recrystallized (1) Uncoated 30 Mil FS-85  
Alloy Sheet from Room Temperature to 2600°F

<u>Test Temperature</u>	<u>UTS PSI (2)</u>	<u>0.2% Offset Y.S.-PSI</u>	<u>% Elongation in 1" Gage</u>	<u>% R.A.</u>
RT	86,900	69,500	21.5	46.7
	85,300	69,200	22.0	54.0
	85,600	69,400	23.0	54.7
400	68,000	53,100	21.2	81.9
	68,000	52,000	20.7	71.5
800	53,300	38,800	17.5	77.8
	54,900	40,900	19.7	92.2
1200	63,100	46,000	15.3	81.2
	61,800	39,100	13.2	77.3
1600	51,800	32,400	17.7	94.7
	53,000	35,300	15.9	64.1
1800	45,000	30,000	17.7	71.5
	44,800	34,600	16.1	72.0
2000	36,000	29,500	28.8	73.3
	36,400	29,400	24.8	77.8
2300	25,200	21,500	39.1	89.2
	24,900	22,000	33.8	86.4
2500	17,400	16,300	50.4	96.1
	16,500	15,600	57.3	92.5
2600	14,700	14,300	78.1	96.7
	14,500	14,100	80.2	97.5

(1) Recrystallized - 1 hour at 2300°F - Vacuum

(2) Strain Rate - 0.020 in/in/min

TABLE 10

Tensile Properties of Pfaunder Coated 30 Mil FS-85 Alloy  
Sheet (1) Tested in Air from RT-2600°F

Test Temperature-°F	UTS PSI (2)	0.2% Offset Y.S. PSI (3)	% Elongation in 1" Gage	% R.A.
RT	87,800	67,900	18.5	19.1
	91,400	73,500	11.0	4.7
	89,400	69,000	20.7	26.1
400	59,400	44,400	19.8	19.8
	64,100	47,700	20.6	20.6
	66,600	49,300	20.7	20.7
800	50,700	35,900	16.5	55.9
	51,200	35,900	14.8	70.2
	50,900	35,400	16.0	50.4
1200	39,100	32,500	4.2	4.0
	39,100	31,600	4.8	5.1
	38,800	31,900	4.5	25.5
1600	42,400	30,100	4.0	16.0
	41,900	31,600	4.3	15.3
	41,800	31,400	4.5	11.4
1800	40,300	28,500	6.9	8.8
	40,200	28,500	6.6	11.4
	41,100	29,300	7.0	23.2
2000	31,900	24,700	11.2	27.3
	33,700	25,600	11.2	17.3
	34,400	26,600	8.2	12.9
2300	24,800	20,500	48.4	34.8
	23,800	20,200	28.4	30.3
	25,800	20,000	25.7	22.2
2500	19,200	16,900	28.0	31.3
	19,200	16,500	31.7	31.9
	19,500	17,200	21.6	31.9
2600	16,400	14,900	43.8	32.8
	16,500	15,300	32.1	46.5
	15,600	14,400	31.5	45.6

(1) Recrystallized in vacuum for 1 hour at 2300°F prior to coating

(2) Strain rate = 0.020 in/in/min

(3) Ultimate and yield strength calculations based on original  
uncoated specimen dimensions



TABLE 11

Tensile Properties of TRW Cr-Ti-Si Coated 30 Mil FS-85 Alloy  
Sheet (1) Tested in Air from RT-2600°F

<u>Test Temperature</u>	<u>UTS PSI (2)</u>	<u>0.2% Offset Y.S. PSI (3)</u>	<u>% Elongation in 1" Gage</u>	<u>% R.A.</u>
RT	79,200	64,200	26.6	58.1
	79,300	62,900	28.0	75.9
	79,400	64,000	27.9	59.4
400	59,400	44,800	22.1	22.1
	63,600	49,100	21.2	21.2
	63,000	47,800	23.2	23.2
800	48,600	35,100	14.8	49.8
	50,200	36,200	16.9	51.5
	51,300	37,000	16.0	46.6
1200	35,700	31,000	5.7	4.7
	36,400	32,600	4.7	11.8
	35,100	31,400	4.4	2.7
1600	35,700	29,500	3.2	2.0
	35,100	29,600	3.3	6.2
	35,500	30,100	2.9	9.0
1800	38,100	26,600	7.5	15.9
	37,800	26,500	6.9	12.1
	36,600	25,800	7.7	19.0
2000	30,800	23,300	20.2	15.2
	30,500	23,200	15.3	17.6
	31,400	23,900	16.6	14.3
2300	21,400	17,400	40.0	24.7
	20,500	16,600	28.5	27.1
	20,700	16,600	27.0	25.2
2500	15,600	14,400	36.3	42.0
	17,400	15,600	45.9	44.3
	17,000	15,200	20.2	58.1
2600	14,800	14,100	100.6	71.6
	14,300	13,400	49.3	63.5
	14,900	13,600	91.6	60.0

(1) Recrystallized in vacuum for 1 hour at 2300°F prior to coating

(2) Strain rate = 0.020 in/in/min

(3) Ultimate and yield strength calculations based on original  
uncoated specimen dimensions

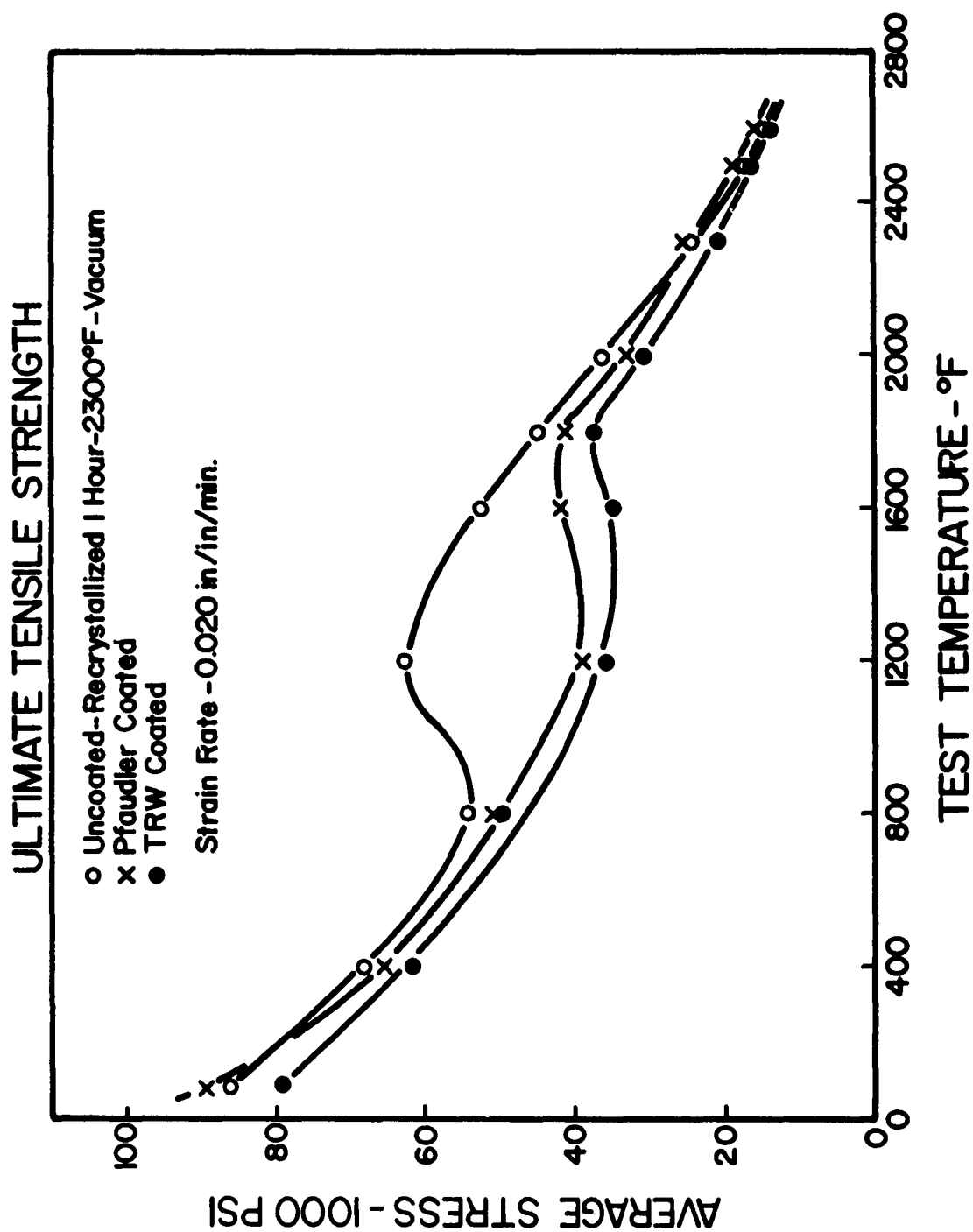


Figure 37 Ultimate Tensile Strength of Uncoated and Coated 30 Mil FS-85 Alloy Sheet from Room Temperature to 2600°F

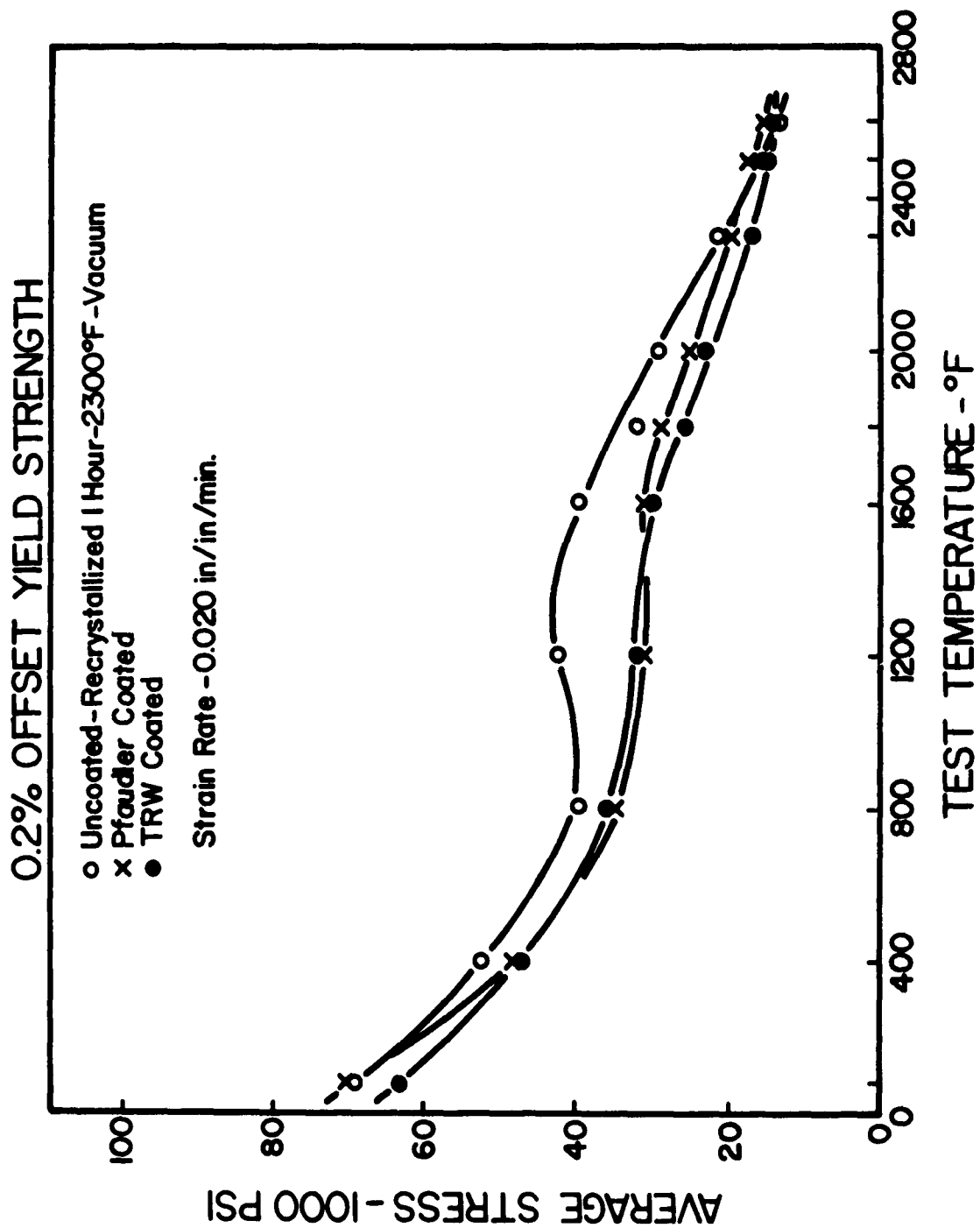


Figure 38 0.2% Offset Yield Strength of Uncoated and Coated 30 Mil FS-85 Alloy Sheet from Room Temperature to 2600°F

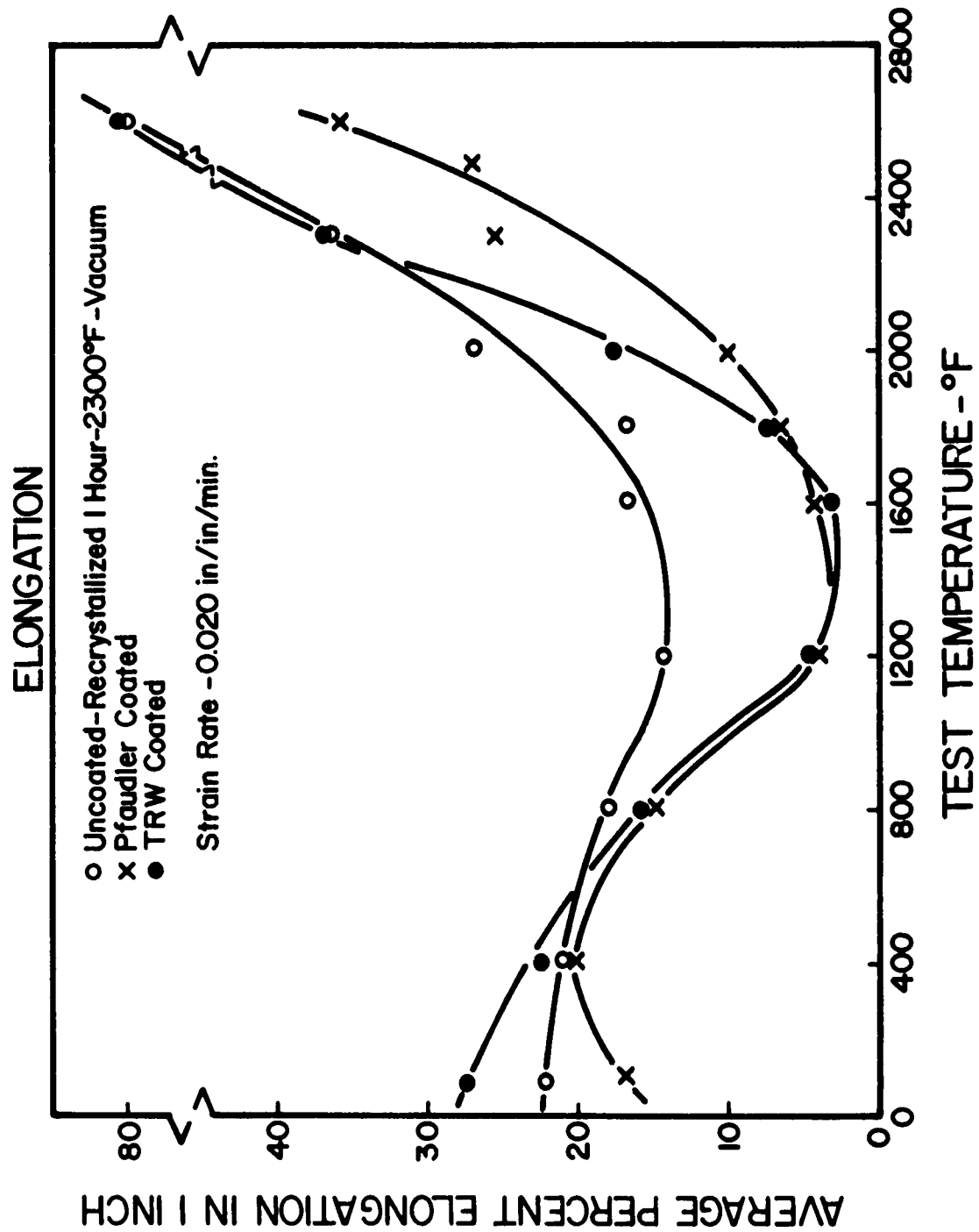


Figure 39 Tensile Elongation of Uncoated and Coated 30 Mil FS-85 Alloy Sheet  
Tested from Room Temperature to 2600°F

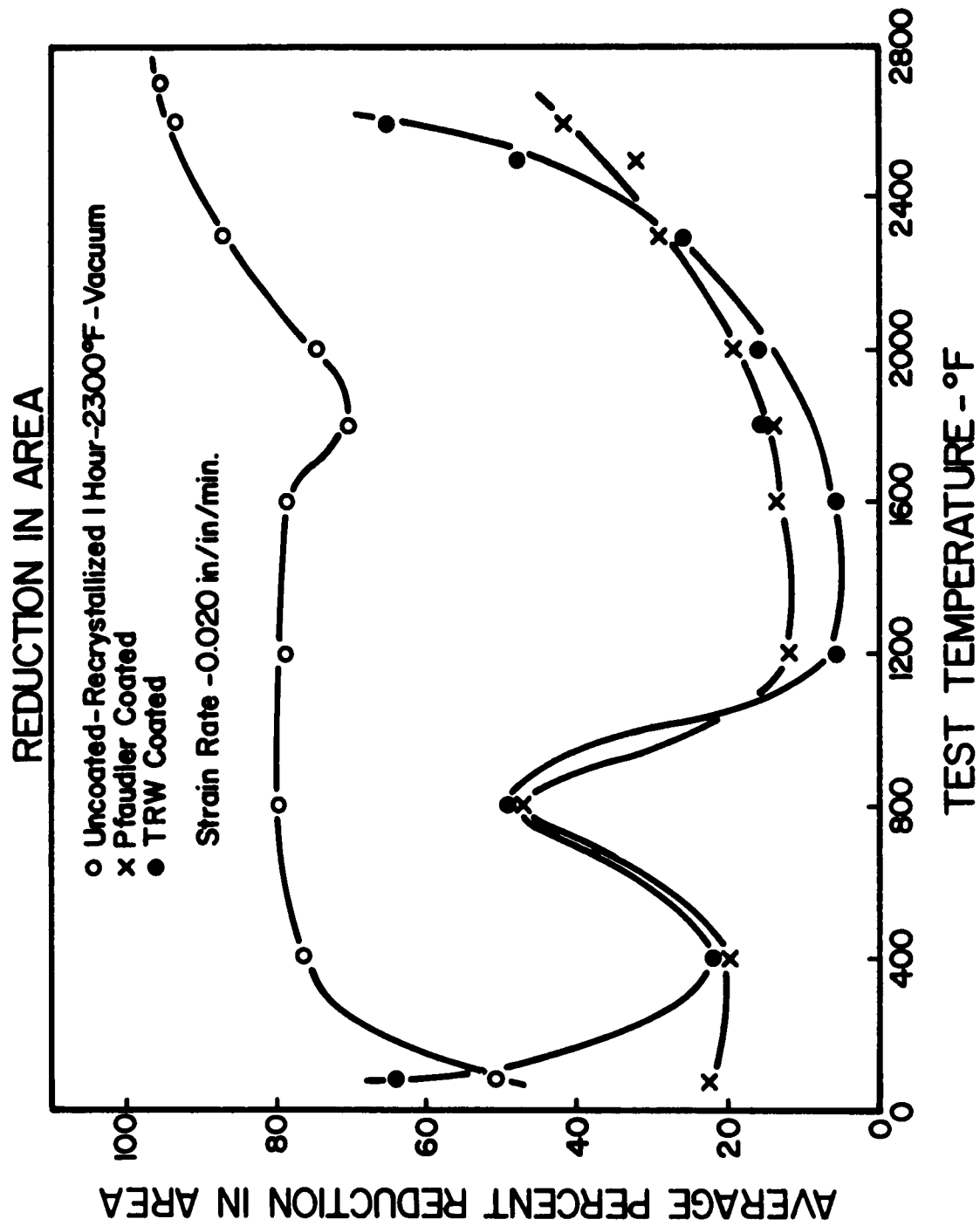


Figure 40 Reduction in Area of Uncoated and Coated 30 Mil FS-85 Alloy Sheet  
Tensile Tested from Room Temperature to 2600°F

uncoated FS-85 alloy was also a region of minimum base metal ductility for both the uncoated and coated material, as evidenced by both the elongation and reduction in area measurements. This change in the characteristics of the strength and ductility curves resulting from the coating may be attributed to both the coating application thermal treatment and to the presence of a brittle surface layer. Cracks produced in the coating after small amounts of deformation would impose severe surface notches in the gage section, in the vicinity of which there would be effectively higher strain rates than that experienced by the uncoated material during uniform deformation. This could result in the propagation of cracks through the substrate reducing the overall ductility. However, if this alteration in properties was totally a notch effect, it is not clear why the brittle behavior of the coated sheet was exhibited in the 1200-1800°F temperature range and not at the lower temperatures where notch effects produced by stress cracks in the coating should be more marked. This behavior tends to indicate the coated substrate became more notch sensitive at the intermediate test temperatures than at lower temperatures.

Additional tensile tests were conducted to factor out the contributions of the coating heat treatment, the brittle coating layer and the strain aging phenomenon at the 1200°F test temperature. The coating layer was completely removed from tensile specimens coated by both Pfaudler and TRW and tensile tests were made in vacuum at 1200°F. These data are reported in Table 12. The substrate ductility, represented by properties of the original uncoated sheet, was completely restored at 1200°F by removal of both the Pfaudler and TRW coatings. Little change was observed in the yield strength as a result of removing the coatings, however, there was observed approximately a 25% increase in the ultimate strength.

The coating heat treatment, therefore, can account for a 20-25% reduction in the ultimate strength of the recrystallized FS-85 alloy sheet and a slight increase in ductility at 1200°F. However, the presence of the brittle coating layer and resulting notch effect contributed an additional 15% reduction in ultimate strength and drastically reduced the composite ductility. At lower temperatures (room temperature to 800°F) the nearly comparable properties of the coated and uncoated sheet indicate the FS-85 alloy was apparently less sensitive to surface and microstructural effects. At 2000°F and above the increased plasticity of the coatings and base metal permitted appreciable coating and substrate deformation prior to the initiation and propagation of cracks through the coatings.





TABLE 12

Tensile Properties of FS-85 at 1200°F Uncoated,  
as Coated, and After Coating Removal

<u>Specimen Condition</u>	<u>UTS PSI(3)</u>	<u>0.2% Offset Y.S. PSI</u>	<u>% Elongation in 1" Gage</u>	<u>% R.A.</u>
Uncoated(Average) <sup>(1)</sup>	62,500	42,500	14.2	79.2
<u>Coated(Average)</u> <sup>(2)</sup>				
Pfaudler	39,000	32,000 <sup>(4)</sup>	4.5	11.5
TRW	35,700	31,700 <sup>(4)</sup>	4.9	9.7
<u>Coating Removed</u> <sup>(1)</sup>				
Pfaudler	49,400	30,300 <sup>(5)</sup>	13.2	80.3
	49,400	30,900	16.2	87.8
TRW	45,600	26,600 <sup>(5)</sup>	19.4	84.5
	47,700	28,800	17.4	87.8

(1) Tested in vacuum

(2) Tested in air

(3) Strain Rate - 0.020 in/in/min

(4) Strength calculations based on original uncoated specimen  
dimensions

(5) Strength calculations based on measured specimen dimensions  
after coating removal

Thus the properties of the uncoated and coated sheet were again nearly comparable.

It is important to note that both coating-base metal systems exhibited nearly comparable mechanical properties over the entire temperature range investigated. Figure 41 shows photomicrographs of the as coated substrate microstructures of the Pfadler and TRW coated FS-85 sheet, indicating a slightly smaller grain size from the lower temperature Pfadler coating treatment.

In order to assure that the radiant heating method and the use of load-extension tensile curves did not introduce questionable experimental error, additional tensile tests were conducted in more conventional tensile testing equipment to corroborate the previously reported mechanical properties. Tensile tests were made at 800, 1200 and 1600°F with TRW coated FS-85 alloy specimens using a gage extensometer and a resistance heated furnace. The mechanical properties determined in these tests were comparable in all cases with those obtained using the radiant heating equipment.

Additional room temperature tensile tests were also conducted on uncoated and both Pfadler and TRW coated specimens using a Baldwin tensile machine, an extensometer, and a strain paced strain rate of 0.005 inches per inch per minute (as recommended by the MAB Panel) until fracture. Strength and ductility values were again comparable with those obtained using the Instron machine at the higher (from a hot testing standpoint-more desirable) strain rate of 0.020 inches per inch per minute. These data are tabulated in Table 13.

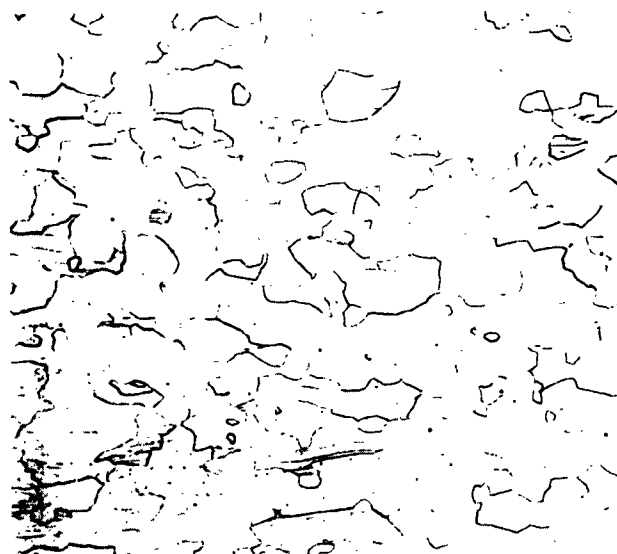
#### 5.6 Coating Deformation Tolerance

In designing to the mechanical properties of a coated structural alloy, other factors peculiar to the coating-base metal system must be considered in addition to the base metal properties and coating protective life. Particularly important is the tolerance of the coating-base metal system to composite deformation without the loss of coating protective characteristics. The ability of the surface alloy layer to undergo elastic and plastic deformation is a function of the temperature, and may also be influenced by the deformation characteristics of the integrally bonded substrate material.

The purpose of this investigation was to determine the prestrain tolerance of coated FS-85 alloy at various temperatures without the loss of protective properties as evidenced by subsequent cyclic oxidation exposure. Pfadler and TRW coated FS-85 alloy Type A



Pfaudler Coated



TRW Coated

Figure 41 As Coated Microstructures of Pfaudler and TRW Coated  
FS-85 Alloy 250X

TABLE 13  
Tensile Properties of Uncoated and Pfaudler and TRW Coated FS-85 Determined  
Using Different Tensile Testing Techniques

Test Temperature-°F	Strain Rate In./In./Min	Coating	Ultimate Strength PSI (4)	0.2% Offset Yield Strength-PSI	% Elongation in 1 In.	% R.A.
RT	0.005 (1)	Uncoated	85,700	69,300	24.7	52.2
RT	0.005	Pfaudler	91,400	70,900	22.9	31.2
RT	0.005	TRW	81,900	65,000	25.7	48.2
800 (3)	0.020 (2)	TRW	51,300	36,500	16.3	57.8
1200	0.020	TRW	37,000	31,600	3.3	1.6
1600	0.020	TRW	33,700	30,000	3.9	7.9

- (1) Strain Rate = 0.005 in./in./min = Baldwin tensile machine = Extensometer  
 (2) Strain Rate = 0.020 in./in./min = Instron tensile machine = Extensometer  
 (3) Heated in resistance wound Marshall tube furnace = 800, 1200, and 1600°F  
 (4) Strength calculations based on original uncoated specimen dimensions



tensile specimens were prestrained in the Instron tensile test machine at various temperatures from RT to 2600°F, using the radiant heating furnace discussed in Section 5.5. A strain rate of 0.020 inches per inch per minute was maintained during both the loading and unloading cycles. At each temperature coated specimens were prestrained at various levels in both the elastic and plastic deformation ranges of the base material. The temperature of each specimen was monitored with a Pt-Pt+10%Rh thermocouple.

The prestrained specimens were then cyclic oxidized in air for 2 hours at 1600 and 2600°F and inspected under 30X magnification for coating failures. The 1600°F exposure temperature was considered to be below the temperature range in which coating selfhealing would be effective, and 2600°F within the temperature range where silicide type coating selfhealing mechanisms are functional. Two hours was selected as an arbitrary exposure limit, and undoubtedly the strain tolerance levels would be a function of the post-oxidation exposure time. Strain induced coating failure was defined as the evidence of coating failure and the growth of columbium oxide within the area of the specimen gage section.

Table 14 presents the results of this study, indicating the levels of prestrain and the post-oxidation test observations. Table 15 lists the mean limits of prestrain which did not produce post-oxidation coating failure and these values are plotted as a function of the prestrain temperature in Figures 42 and 43.

The prestrain levels were selected arbitrarily in an effort to bracket the prestrain tolerance of the two coatings at the various prestrain temperatures. The tensile load-extension curves plotted continuously by the Instron test machine were utilized in estimating the specimen deformation. Automatic termination of the crosshead travel was preset for the desired prestrain deformation and the tensile load was released at the 0.020 in/in/min. strain rate. Each prestrained specimen was then accurately measured on a comparator to determine the percent plastic deformation within the gage section. The approximate elastic deformation determined from the Instron load-extension curve was added to the measured plastic deformation and these values are reported in Table 14 as the total prestrain. Table 15 lists the prestrain tolerance obtained by calculating the mean value between a prestrain level which produced a post-oxidation coating failure and the nearest prestrain level which did not produce coating failure. Preliminary data reported for this evaluation test included the total prestrain extension values selected from the Instron plot of crosshead travel, and did not include the correction based on comparator measurements of gage section plastic deformation.

TABLE 14

Tensile Prestrain + Oxidation Test Results of  
Pfaudler and TRW Coated FS-85 Alloy

Test Temperature °F	Average Proportional Limit % Strain <sup>(4)</sup>	TRW Coating			Pfaudler Coating		
		Prestrain Level	Condition After 2 Hours		Prestrain Level	Condition After 2 Hours	
		% Total Strain <sup>(1)</sup>	Oxidation		% Total Strain	Oxidation	
			1600°F	2600°F		1600°F	2600°F
R.T.	2.2	2.8	-	*(2)	2.1	-	*
		3.0	*	-	2.5	*	-
		3.5	-	*	2.5	*	-
		4.1	-	*	2.8	-	*
		4.3	*	-	3.1	-	F
		4.9	-	F(3)	3.3	-	F
		6.0	-	F	4.1	F	-
		6.3	*	-	4.5	F	-
		6.4	F	-			
		6.5	-	F			
400	1.8	2.2	-	*	2.0	*	-
		2.4	*	-	2.1	-	*
		3.6	-	*	2.3	-	*
		3.7	*	-	3.1	*	F
		3.8	-	*	3.1	F	-
		4.4	F	-	3.3	F	-
		4.9	*	-	3.8	F	-
		5.3	-	*	4.7	-	F
		5.7	-	F			
		6.6	-	F			
800	1.6	1.9	*	-	0.9	-	*
		2.2	-	*	1.7	-	F
		3.7	-	*	2.1	-	F
		3.9	-	*	2.5	-	F
		4.1	*	-	2.4	-	-
		5.3	-	F	2.5	-	F
		6.2	*	-	2.9	F	-
		7.4	*	-	3.0	F	-
		8.5	*	-	3.3	F	-

(1) Total strain - elastic + plastic strain

(2) \* - Indicates no evidence of coating failure in prestrained gage section

(3) F - Denotes coating failure in prestrained gage section

(4) Proportional limit - obtained from load-extension curve plotted by Instron machine



TABLE 14 (CONTINUED)

Test Temperature °F	Average Proportional Limit % Strain <sup>(4)</sup>	TRW Coating			Pfaudler Coating		
		Prestrain Level	Condition After 2 Hours		Prestrain Level	Condition After 2 Hours	
		% Total Strain <sup>(1)</sup>	Oxidation		% Total Strain	Oxidation	
			1600°F	2600°F		1600°F	2600°F
1200	1.4	1.4	*	-	1.2	-	*
		1.7	-	*	1.3	*	-
		3.4	-	*	1.5	*	-
		3.5	*	-	2.0	-	*
		4.1	*	*	2.5	-	F
					2.6	F	-
					3.1	F	-
					3.2	-	F
1600	1.1	2.0	*	*	1.3	-	*
					1.4	-	F
					1.5	*	F
					2.4	F	-
					2.7	F	-
2000	1.1	3.2	-	*	2.2	-	*
		3.6	-	*	4.0	F	-
		4.0	*	*	4.4	*	-
		5.5	*	-	5.1	F	-
		6.2	*	-	5.2	-	*
		7.8	F	-	5.2	-	F
					6.5	F	F
2300	1.1	13.8	*	-	5.7	F	-
		13.9	-	*	8.7	-	*
		16.3	-	*	8.9	F	-
		17.2	-	*	8.9	F	-
		18.4	*	-	11.3	-	*
		18.7	-	F	12.9	-	F
		24.0	*	-	14.1	-	F
		27.6	F	-	14.9	F	-
2600	1.1	34.5	*	-	21.5	-	*
		37.3	-	*	23.8	F	-
					37.2	F	-
					38.5	-	*

- (1) Total strain - elastic + plastic strain  
 (2) \* - Indicates no evidence of coating failure in prestrained gage section  
 (3) F - Denotes coating failure in prestrained gage section  
 (4) Proportional limit - obtained from load-extension curve plotted by Instron machine

TABLE 15

Tolerance of Pfaudler and TRW Coatings on FS-85 Alloy To  
 Deformation at Various Temperatures Without Loss Of  
 2 Hour Oxidation Protection at 1600°F and 2600°F

Prestrain Temperature °F	Average Proportional Limit-% Strain(1)	Limit of Prestrain Without Loss of 2 Hour Protection-% Total Strain			
		TRW		Pfaudler	
		1600°F	2600°F	1600°F	2600°F
R.T.	2.2	6.4	4.5	3.3	3.0
400	1.8	4.1	5.5	3.1	2.7
800	1.6	> 8.5	4.6	2.7	1.3
1200	1.4	> 4.1(2)	> 4.1	2.1	2.3
1600	1.1	> 2.0	> 2.0	2.0	1.4
2000	1.1	7.0	> 4.0	4.2	5.2
2300	1.1	25.8	18.0	< 5.7	12.1
2600	1.1	> 34.5	> 37.3	< 23.8	> 38.5

- (1) Average prestrain value between strain which produced and strain which did not produce post-oxidation coating failure- from Table 14
- (2) Tensile elongation of coated base metal limited prestrain in 1200-1600°F temperature range - see tensile properties, Section 5.5



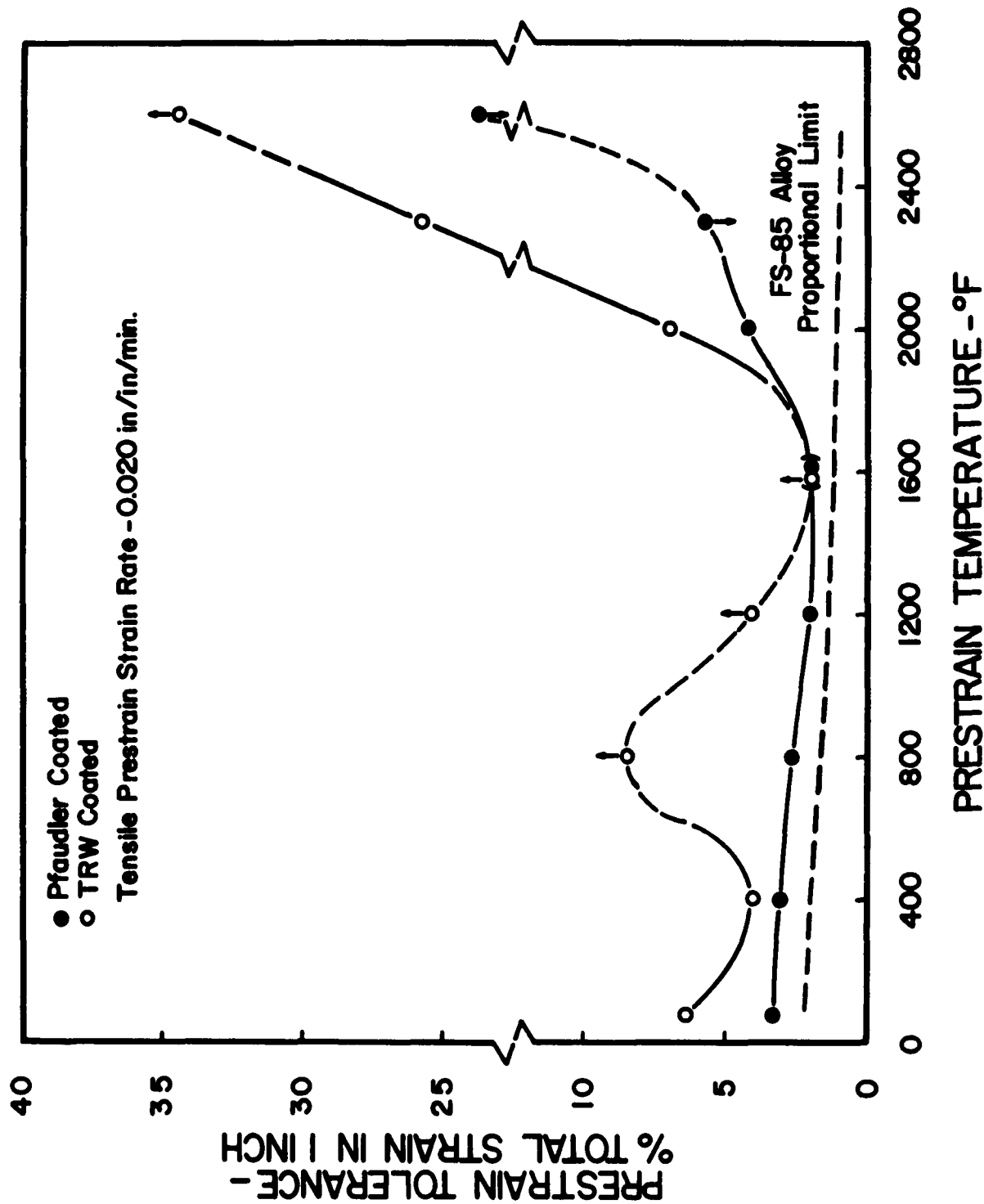


Figure 42 Limit of Coating-Base Metal Deformation at Various Prestrain Temperatures Without Loss of 2 Hour Post Oxidation Protection at 1600°F for Pfaudler and TRW Coated FS-85 Alloy

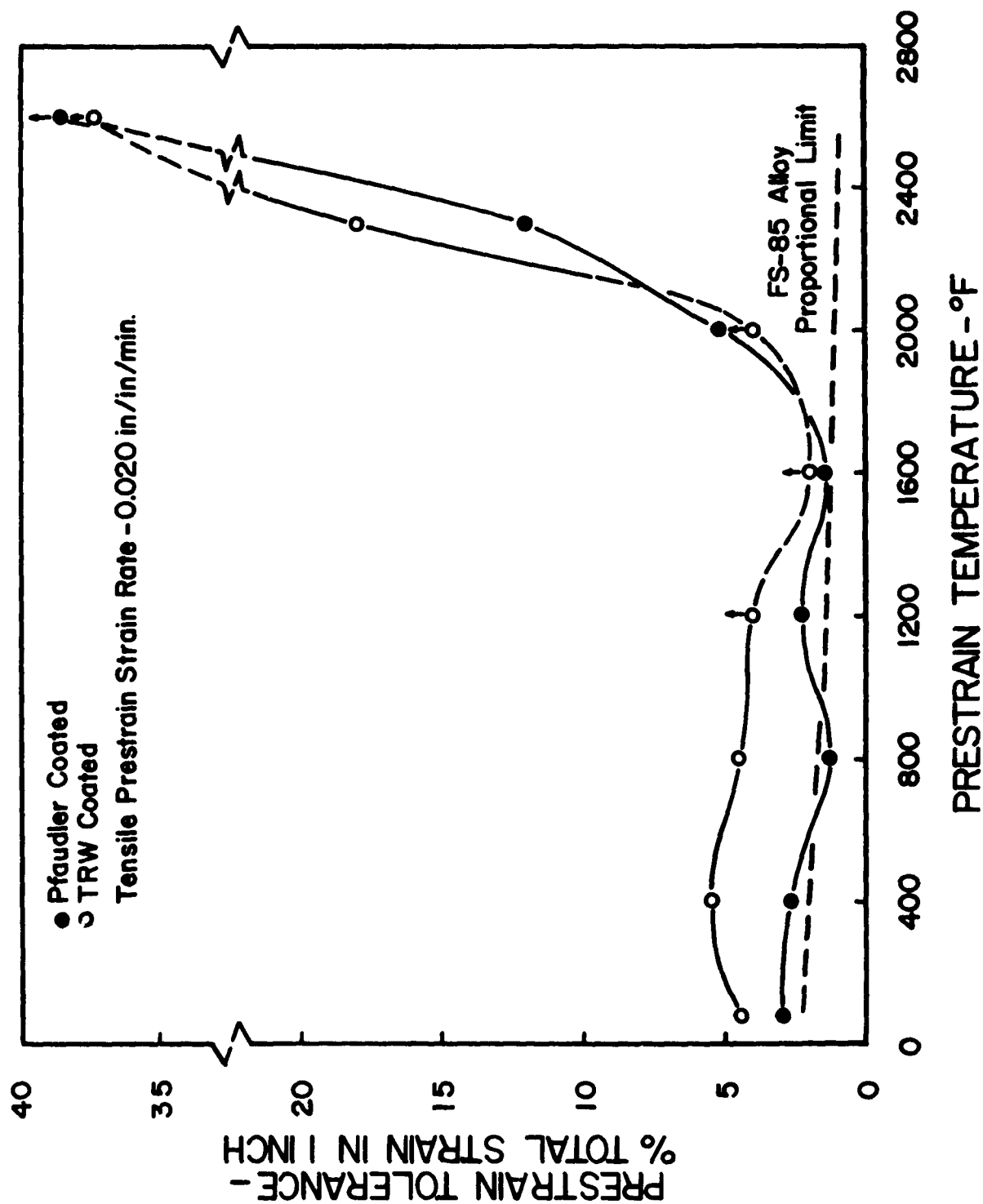


Figure 43 Limit of Coating-Base Metal Deformation at Various Prestrain Temperatures Without Loss of 2 Hour Post Oxidation Protection at 2600°F for Pfautler and TRW Coated FS-85 Alloy



The specimen design for this test was found to be not entirely adequate. Because of the relatively small differential between the cross section of the gage section and the pin hole load bearing surface yielding frequently occurred in the pin hole as well as in the specimen gage section. This yielding coupled with other extensions in the loading system rendered the extension values recorded by the cross-head travel to the set point significantly greater than the actual gage section prestrain. For better testing efficiency and accuracy the specimen design should assure that virtually all deformation recorded for the cross-head movement be confined to the specimen gage section.

In general, under the conditions tested, the TRW coating was capable of withstanding more deformation without the loss of oxidation protection than was the Pfaudler coating. The low base metal ductility in the 800-1600°F temperature range limited the prestrain, and in some cases coating failure was not produced by prestraining at levels just below that which would cause nearly brittle fracture of the substrate. However, the strain tolerance of both coatings also reached minima in this temperature range. Apparently the brittle behavior of the substrate resulted in a more localized deformation pattern, thereby propagating cracks through the coating into the substrate at lower overall measured strain levels. Arrows in the plot of Figures 42 and 43 are utilized to indicate the data points which were not firmly established and these portions of the plotted curve are indicated by a dashed line.

The plasticity of the two coatings greatly increased at temperatures of 2000°F and above, permitting in excess of 35% prestrain at 2600°F without the loss of coating protection. Figure 44 is a photograph showing both Pfaudler and TRW coated FS-85 alloy specimens prestrained 30-40% at 2600°F and post-oxidized for 2 hours at 1600 and 2600°F. Only the Pfaudler coated specimen post-oxidized at 1600°F shows evidence of coating failure.

In general both coatings tolerated more prestrain for subsequent oxidation at 1600°F than for post oxidation at 2600°F. This was not expected since flaws in the coatings could not selfheal at 1600°F, as they could at the higher temperature. At both oxidation temperatures, strain induced coating failures generally occurred along the specimen edges.

The most important point to be emphasized from this data is; however, that with only one exception both the Pfaudler and TRW coatings were prestrained beyond the base metal proportional limit at all temperatures from RT to 2600°F without loss of subsequent 2 hour oxidation protection at 1600 and 2600°F. This is extremely



As Coated



Prestrained 36.0%

Post Oxidation - 2 Hours - 1600°F



Prestrained 37.3%

Post Oxidation - 2 Hours - 2600°F

Pfudler Coated



Prestrained 36.5%

Post Oxidation - 2 Hours - 1600°F



Prestrained 36.2%

Post Oxidation - 2 Hours - 2600°F

TRW Coated

Figure 44 Photograph of Pfudler and TRW Coated FS-85 Alloy Specimens  
Tensile Prestrained at 2600°F and Post Oxidation Tested for  
2 Hours at 1600 and 2600°F



significant since metal structures are designed for service at stress levels below the base metal yield stress. Although coating failures were not observed on many of the prestrained specimens after 2 hours post-oxidation, the ultimate coating protective life was undoubtedly impaired. An investigation of the relative prestrain damage at the various prestrain levels would require considerably more test specimens and a quite lengthy oxidation testing program.

### 5.7 Stress Rupture Tests

Stress rupture testing a coated refractory metal in air evaluates essentially two interrelated time dependent mechanisms, base metal creep and creep deformation of the protective coating. Depending on the exposure temperature the coating will possess a limited deformation tolerance before loss of protection, and this deformation limit will establish a finite stress rupture life of the coating-base metal system. A third factor influencing the stress rupture life of a coating-base metal composite is the presence of coating defects. Obviously these imperfections do not constitute a directly measurable property of the coating-base metal system. However, the occurrence of localized coating failures and the ability of a coating to selfheal these defects while under stress at elevated temperatures are important factors in evaluating the reliability of a protective coating system. Under low stress where base metal creep is negligible, the protective life of a coating and its effectiveness as an oxygen diffusion barrier determine the ultimate failure or rupture life of a coating-base metal system.

In this portion of the design data study the stress rupture properties of Pfudler and TRW coated FS-85 alloy were investigated in air at 1600, 1800, 2000, 2300, 2500 and 2600°F. A minimum of four (4) stress levels were evaluated at each temperature for a maximum stress-oxidation exposure of approximately 150 hours. At 1600-2000°F the stress rupture tests were conducted in conventional Arc Weld stress rupture equipment, using Udimet 700 alloy specimen holders and TRW 1800 alloy specimen pins. The specimen configuration was shown as Type C in Figure 26. Chromel-alumel thermocouples were utilized to control the specimen temperature at  $\pm 3^\circ\text{F}$ , with no measurable temperature gradient evident along the one inch gage length.

At 2300-2600°F the stress rupture tests were conducted in a multiple rack global heated furnace shown in Figure 45. Figure 46 is a photograph of the components of one of two stress rupture loading assemblies. The refractory tube and split support cap were both pure alumina. Cr-Ti-Si coated D-31 and D-41 columbium alloy pins were utilized for supporting the coated columbium alloy specimens.

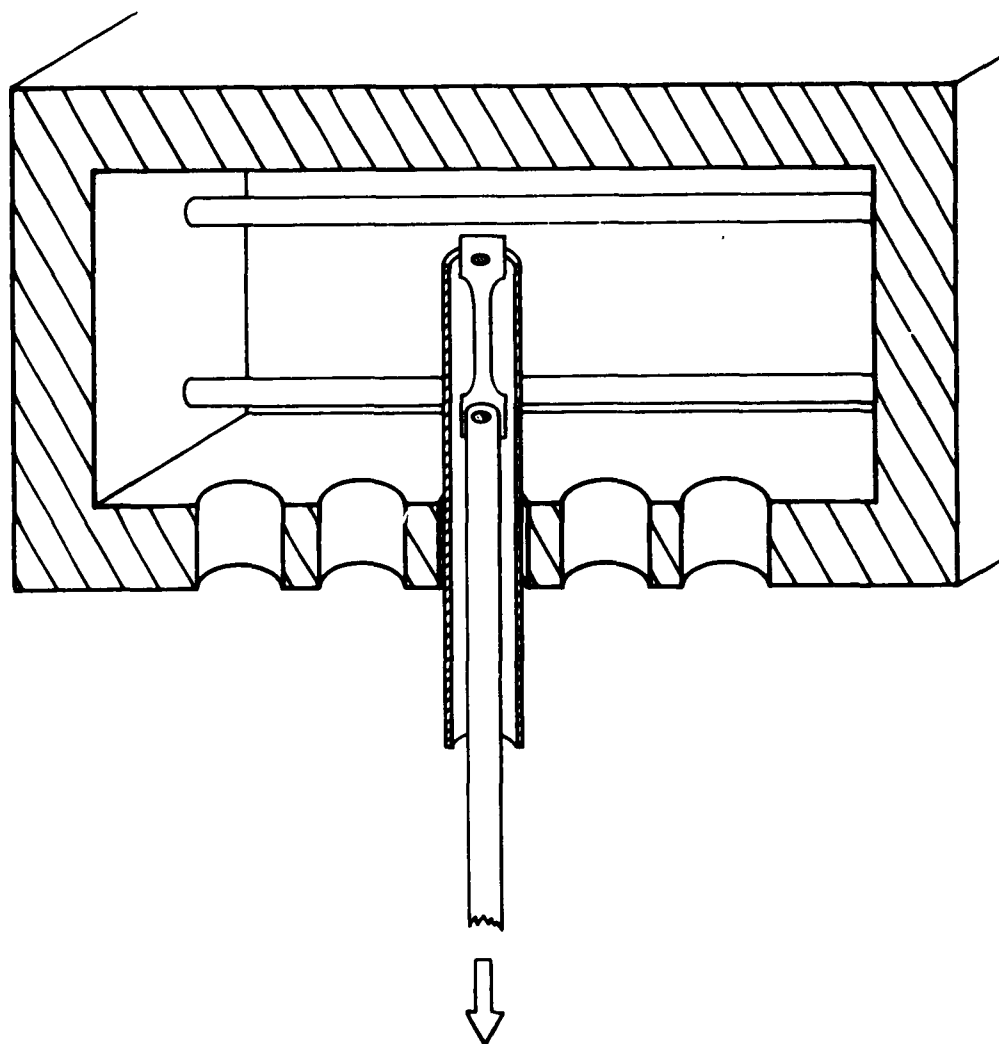


Figure 45 Cross Section of the Hearth of a Globar Heated Multiple  
Rack Stress Oxidation Furnace Approx. 1/3X

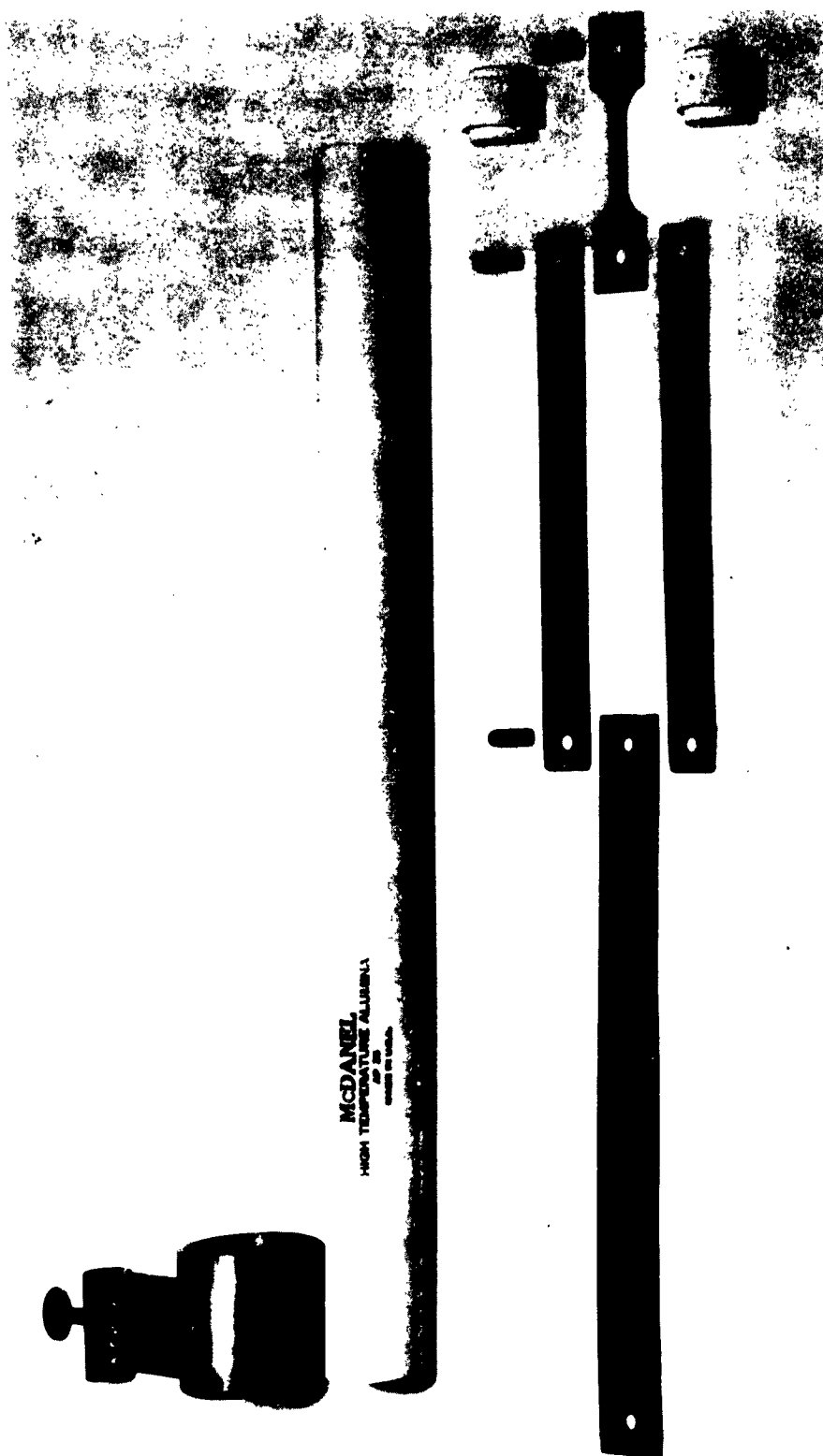


Figure 46 Layout of Stress Rupture Loading Assembly Components for Stress Rupture Testing in Air in the Temperature Range 2300-2600°F Approximately 1/3X

Three loading strap materials were used successfully: pure alumina, coated unalloyed molybdenum and Cr-Ti-Si coated Cb-10Ti-10W alloy sheet. The Cr-Ti-Si coated columbium alloy straps provided the advantages of base metal ductility and remarkable load bearing capability even after appreciable coating failure. These straps were therefore utilized for the majority of the stress rupture tests.

In conducting the stress rupture tests using this apparatus, a specimen was assembled in the tubular loading device outside the furnace, and the assembly was inserted into the hot zone through the furnace base. The load was applied through the strap linkage after the specimen reached temperature. Pt-Pt+10%Rh thermocouples were used to monitor the temperature of each individual specimen.

For the higher stress levels it was necessary to support the specimen load from outside the furnace. This was accomplished by utilizing identical Cr-Ti-Si coated columbium alloy straps and pins, attached to both ends of the test specimen. The furnace was modified to permit loading of the specimen and straps through holes in the roof of the furnace. The load was directly applied through weights suspended from the bottom straps and was supported through the upper straps from an external platform above the furnace.

Table 16 presents the results of the stress rupture study. The stress levels ranged from 20 to 90% of the base metal yield strength, based on tensile test results on uncoated FS-85 alloy sheet (Section 5.5). All tests were terminated after approximately 150 hours of stress oxidation, with the exact time of termination depending upon the test schedule.

Specimen elongation measurements were not made on several specimens after rupture because of gross oxidation at the exposed fracture surface. It was obviously necessary to remove the fractured specimens from the test furnaces immediately after failure.

A serious testing difficulty was encountered at 1600 and 2600°F involving rupture of the test specimens in areas other than in specimen gage sections. At 1600°F the frequent occurrence of pinhole fractures was attributed to localized creep deformation at the pinhole, which resulted in coating failure and ultimately specimen fracture. As pointed out in Section 5.6, both coatings exhibited a minimum strain tolerance on FS-85 alloy at 1600°F. The problem of pinhole failure could be eliminated by utilizing test specimens of smaller gage cross section relative to the load bearing area of the pinhole and grip section.





TABLE 16

Stress Rupture Results of Pfaudler and TRW Coated  
FS-85 Alloy Tested in Air at 1600 - 2600°F

Test Temperature °F	Stress Level		Pfaudler		TRW	
	% of Y.S.	PSI	Hours to Failure	% Elongation	Hours to Failure	% Elongation
1600	40	13540	162.4*(1) 162.9*	0.25 0.25	114.8(2) 163.2* 184.8*	3.3 0.6 0.6
1600	60	20310	* 34.6(2) 59.1(2) 68.2(2) 163.0*	1.0 0.8 2.9 0.0	134.5(2) 146.5* 162.4*	1.1 1.0 1.1
1600	70	23695	15.6(2) 21.8(2) 61.2(2) 68.6(2) 159.7*	0.3 0.1 2.0 0.0 0.2	139.3(2) 145.4* 146.0*	1.9 1.6 1.0
1600	75	25388	1.8 7.6(2) 14.0(2) 32.8(2) 64.1(2)	1.6 3.9 0.0 1.9 7.6	-	-
1600	80	27080	1.5 4.5	1.9 3.3	152.9* 157.4*	9.5 5.5
1600	90	30465			0.0 11.7 167.8*	2.9 2.9 3.6
1800	40	12920	141.4 162.5*	4.9 1.3	145.0 164.3*	2.7 1.6
1800	60	19380	1.9 162.8* 163.0*	- (3) 3.7 2.3	145.4* 165.4*	5.7 6.4

(1) \* - Test discontinued - no rupture

(2) Specimen failed in pin hole or grip section

(3) Gross oxidation \* no elongation measurement

(4) Specimen cycled to room temperature during test

TABLE 16 (CONTINUED)

Stress Rupture Results of Pfaudler and TRW Coated  
FS-85 Alloy Tested in Air at 1600 - 2600°F

Test Temperature °F	Stress Level		Pfaudler		TRW	
	% of Y.S.	PSI	Hours to Failure	% Elongation	Hours to Failure	% Elongation
1800	70	22610	145.4* 159.7*	9.9 10.3	114.9 138.5	13.2 4.0
1800	75	24225			53.4 92.1	0.4 14.8
1800	80	25840	88.1 90.8 134.5	33.7 26.9 30.8	4.1 39.3 41.7	7.1 13.6 13.6
1800	85	27455	0.0 1.0	2.8 4.2	- -	- -
2000	40	11780	146.0* 146.8*	17.7 2.5	58.1 58.7 145.1*	3.4 4.5 3.2
2000	60	17670	102.8 145.4	29.2 56.6	10.5 102.8 123.7 160.6*	26.8 52.5 77.8 58.2
2000	65	19143	13.2 57.4 161.7*	34.7 41.0 36.7	4.6 29.1 111.0	22.4 31.6 - (3)
2000	70	20615	29.6 46.6	46.2 42.1	6.2 19.5 38.6	25.0 31.6 68.4
2000	80	23560	7.2 20.5	47.4 46.8	5.3 5.3	28.5 30.8

- (1) \* - Test discontinued - no rupture  
(2) Specimen failed in pin hole or grip section  
(3) Gross oxidation - no elongation measurement  
(4) Specimen cycled to room temperature during test



TABLE 16 (CONTINUED)

Stress Rupture Results of Pfaudler and TRW Coated  
FS-85 Alloy Tested in Air at 1600 - 2600°F

Test Temperature °F	Stress Level		Pfaudler		TRW	
	% of Y.S.	PSI	Hours to Failure	% Elongation	Hours to Failure	% Elongation
2300	40	8700	114.6	-(3)	145.8*	30.5
			115.4	49.9	146.6*	33.6
2300	50	10875	72.1	-(3)	53.3	104.3
			73.4	-(3)	55.8	113.4
2300	60	13050	15.9	92.2	14.7	85.4
			26.0	88.7	17.5	114.0
			36.8	53.0		
2300	70	15225	3.5	50.6	4.3	94.1
			16.8	68.7	4.7	90.3
			19.9	45.8		
2500	20	3190	19.2	-(3)	147.0*	4.3
			20.8	-(3)	159.0*	5.3
2500	40	6380	6.8	-(3)	48.5 <sup>(4)</sup>	11.1
			13.5 <sup>(4)</sup>	5.0	58.3 <sup>(4)</sup>	-(3)
2500	50	7975	24.0	21.6	57.6	-(3)
			33.9	-(3)	63.6	75.8
2500	60	9570	8.6	-(3)	21.0 <sup>(4)</sup>	67.4
			9.2 <sup>(4)</sup>	-(3)	23.8 <sup>(4)</sup>	75.2
2600	20	2840	19.6	-(3)	21.5 <sup>(2)</sup>	0.5
			60.2 <sup>(2)</sup>	1.2	50.8 <sup>(2)</sup>	3.7
			106.6	-(3)	86.5 <sup>(2)</sup>	3.2
2600	40	5680	19.1	18.2	98.7 <sup>(2)</sup>	54.5
			44.5	-(3)	105.5	-(3)
2600	50	7100	11.7	-(3)	3.6 <sup>(2)</sup>	5.0
			17.9	23.3	26.0 <sup>(2)</sup>	-(3)
					67.4	44.2
2600	60	8520	4.1 <sup>(2)</sup>	16	14.3 <sup>(2)</sup>	42.4
			13.4	-(3)	15.7	82.5

(1) \* - Test discontinued - no rupture

(2) Specimen failed in pin hole or grip section

(3) Gross oxidation - no elongation measurement

(4) Specimen cycled to room temperature during test

At 2600°F specimen failures in the grip section were the result of catastrophic coating failure. The stress rupture furnace design placed the specimen grip sections and pins in close proximity to the furnace heating elements. In maintaining 2600°F along the specimen gage section, the grip section and Cr-Ti-Si coated columbium alloy support components undoubtedly experienced higher temperatures which were well above the melting point of columbium oxide. The initiation of a coating failure in the test specimen or in the coated columbium alloy support materials, or reaction between the coatings and refractory alumina support straps resulted in melting and premature specimen failure in the grip section and not in the gage section.

In addition to these types of premature specimen failure, there were also a number of erratic tests in which fracture did occur within the specimen gage section. In the case of the Pfaudler coating it was pointed out previously that coating thickness varied from 1.5-3.2 mils on individual specimens. This would account for some inconsistency in coating protective performance. It is also indicated in Table 16 that several specimens tested at 2500°F were unintentionally cycled to room temperature during the test as a result of loading assembly failures that required reloading specimens with new straps and pins. A comparison of the results of these tests with those of non-thermal cycled specimens clearly indicated that thermal cycling was detrimental to the stress rupture lives of the coated systems. Still another factor contributing to premature stress rupture failures was the presence of coating defects. A coating defect could either cause coating failure immediately upon exposure or could result in localized coating deformation during creep, both resulting in localized oxidation and premature fracture. Thus the stress rupture lives of the individual coated FS-85 alloy specimens can be related to a number of coating failure mechanisms, some of which reflect a definite measure of the coating protective reliability.

One of the most significant results of the stress rupture study is evident from a comparison of the stress rupture lives of the two coating-base metal systems, particularly at the higher temperatures, with the cyclic oxidation test results presented in Section 5.3. At stress levels where creep was appreciable, but not excessive, both coatings were more protective on FS-85 alloy under the non-thermal cycle, stress rupture conditions than under the conditions of periodic cycling to room temperature. Both coatings on FS-85 alloy were apparently susceptible to crack propagation as a result of rapidly imposed thermal stresses, whereas their tolerance for uniform creep deformation was appreciable at 1800°F and above.



Figures 47 and 48 are graphical presentations of the stress rupture data for coated FS-85 alloy. The values plotted are not exact averages of all the data, but are average values of those tests considered representative of the stress oxidation characteristics of the two coating-base metal systems. The erratic tests, for which rupture was attributed to other than stress induced failure in the gage section, were omitted. The values plotted on the curves then represent all valid tests for stress levels which produced rupture in less than 150 hours, and an additional point for the highest stress level which did not produce fracture in 150 hours. From these curves, 10 and 100 hour stress to rupture values were determined for each test temperature. These values are tabulated in Table 17. In general, the stress rupture properties of the two coating - FS-85 alloy composites were nearly comparable over the 1600-2600°F temperature range.

At temperatures up to 2000°F the base metal creep rate was relatively low at stress levels less than 60% of the base metal yield strength. However, the creep rate increased significantly at 2300°F and above, resulting in as much as 100% specimen elongation prior to rupture. It is not apparent in these cases whether rupture was the result of a strain induced coating failure, or if fracture was initiated internally as a result of base metal creep. Figure 49 shows photographs of Pfaudler and TRW coated specimens stress rupture tested at 2300 and 2600°F. These specimens were removed from the furnace immediately after fracture. Note the extreme oxidation damage suffered by the Pfaudler coated specimen tested at 2600°F prior to rupture in the gage section. The initiation of coating failure and the stress rupture life are obviously not factors which can be closely related.

The tolerance of the coatings for deformation at 1800°F and above and the extreme amount of creep required for rupture at these temperatures points up the question of which is the more significant property, stress to rupture or stress to produce a limited amount of creep and still retain coating protective properties. These data clearly indicate that both coating-base metal systems were capable of tolerating creep deformation far in excess of that applicable in design considerations without rupture or the evidence of coating failure.

Figures 50 and 51 are photographs of representative Pfaudler and TRW coated FS-85 alloy specimens which did not rupture after approximately 150 hours of stress oxidation exposure. Room temperature tensile tests were conducted on these and other specimens, which did not fracture in the stress rupture test, to assess the effect of

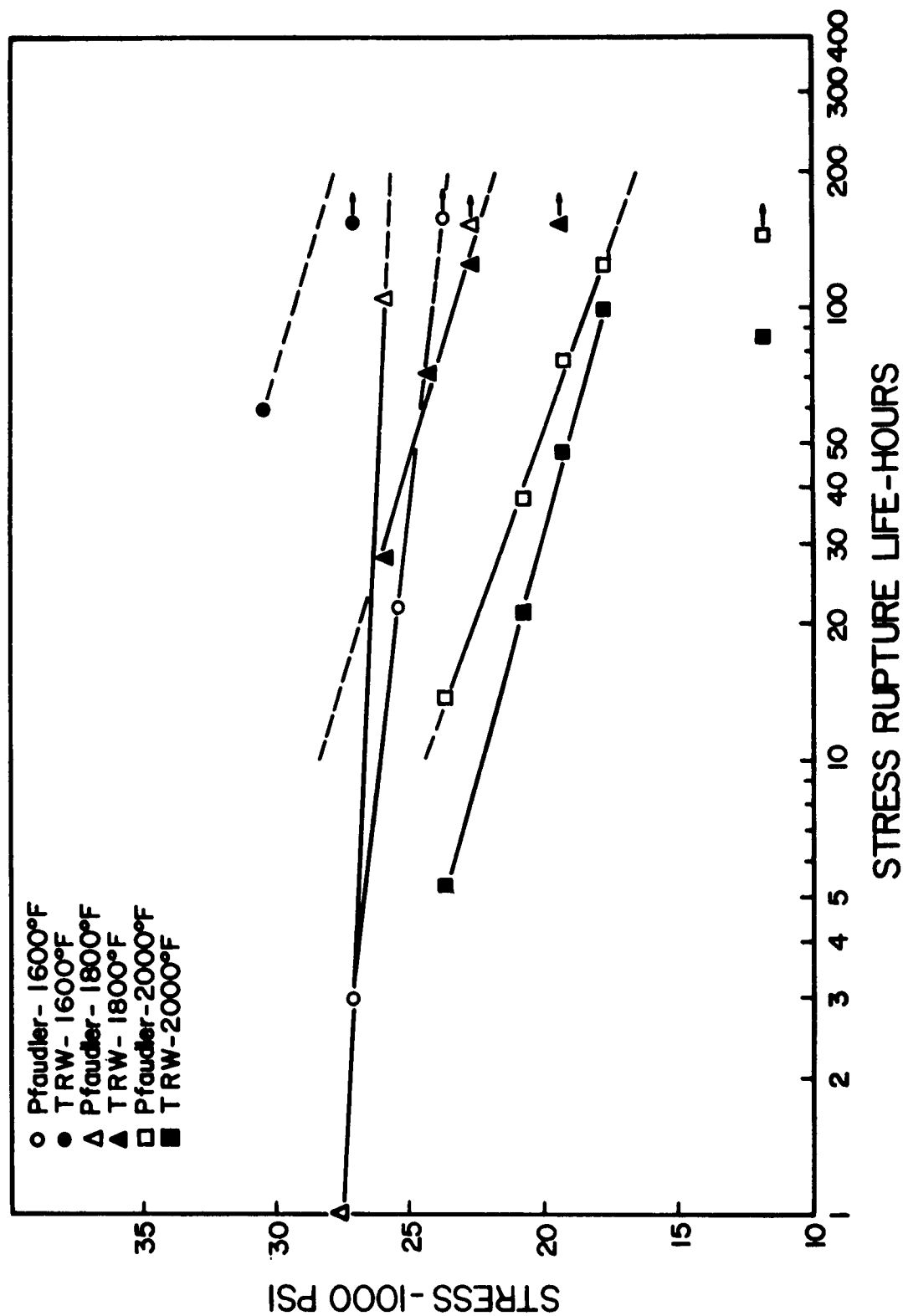


Figure 47 Stress Rupture Life as a Function of Stress for Pfaudler and TRW Coated FS-85 Alloy Tested in Air at 1600-2000°F

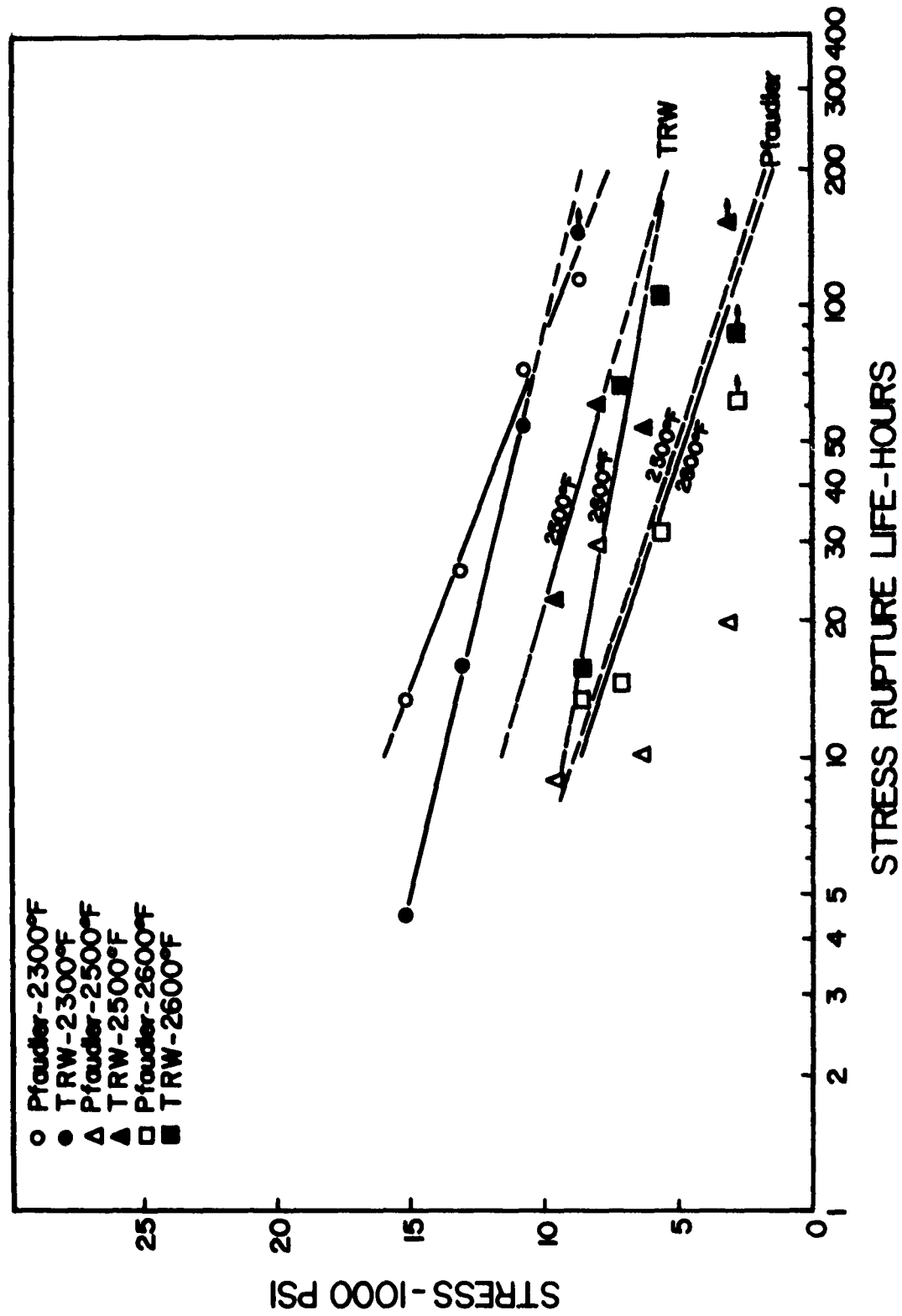


Figure 48 Stress Rupture Life as a Function of Stress for Pfaudler and TRW Coated FS-85 Alloy Tested in Air at 2300-2600°F

TABLE 17

Stress to Rupture in 10 and 100 Hours at Temperatures  
 from 1600 to 2600°F for Pfaudler and TRW Coated FS-85  
 Alloy Tested in Air

Test Temperature °F	Stress to Rupture - PSI <sup>(1)</sup>			
	Pfaudler Coated		TRW Coated	
	10 Hours	100 Hours	10 Hours	100 Hours
1600	26100	24200	30500	29300
1800	26700	25900	28400	23300
2000	24500	18300	22300	17600
2300	16000	9500	13800	9800
2500	8900	3400	11600	6800
2600	8600	3100	9300	6250

(1) Stress levels determined from plotted stress rupture life  
 data in Figures 47 and 48





As Coated



2300°F  
15.9 Hrs.

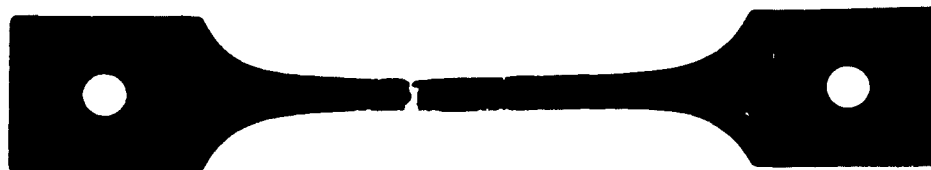
13,050 PSI  
92.2% Creep



2600°F  
106.6 Hrs.

2840 PSI

Pfaudler Coated



2300°F  
17.5 Hrs.

13,050 PSI  
114.0% Creep

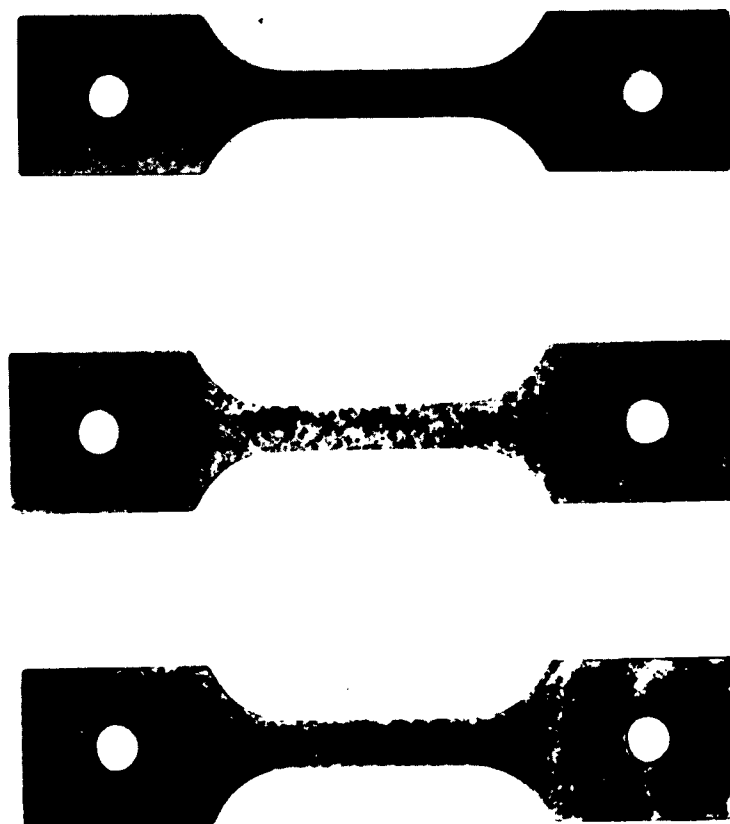


2600°F  
98.7 Hrs.

5680 PSI  
54.5% Creep

TRW Coated

Figure 49 Pfaudler and TRW Coated FS-85 Alloy Specimens Stress Rupture Tested in Air at 2300 and 2500°F 1X



Temperature - °F	1600	1800	2000
Stress - PSI	23,695	22,610	11,780
Exposure - Hours	159.7	159.7	146.0
% Creep Elongation	0.2	10.3	17.7

Figure 50 Pfaudler Coated FS-35 Alloy Stress Rupture Specimens Stress Oxidation Tested  
Without Failure in Air at 1600-2000°F

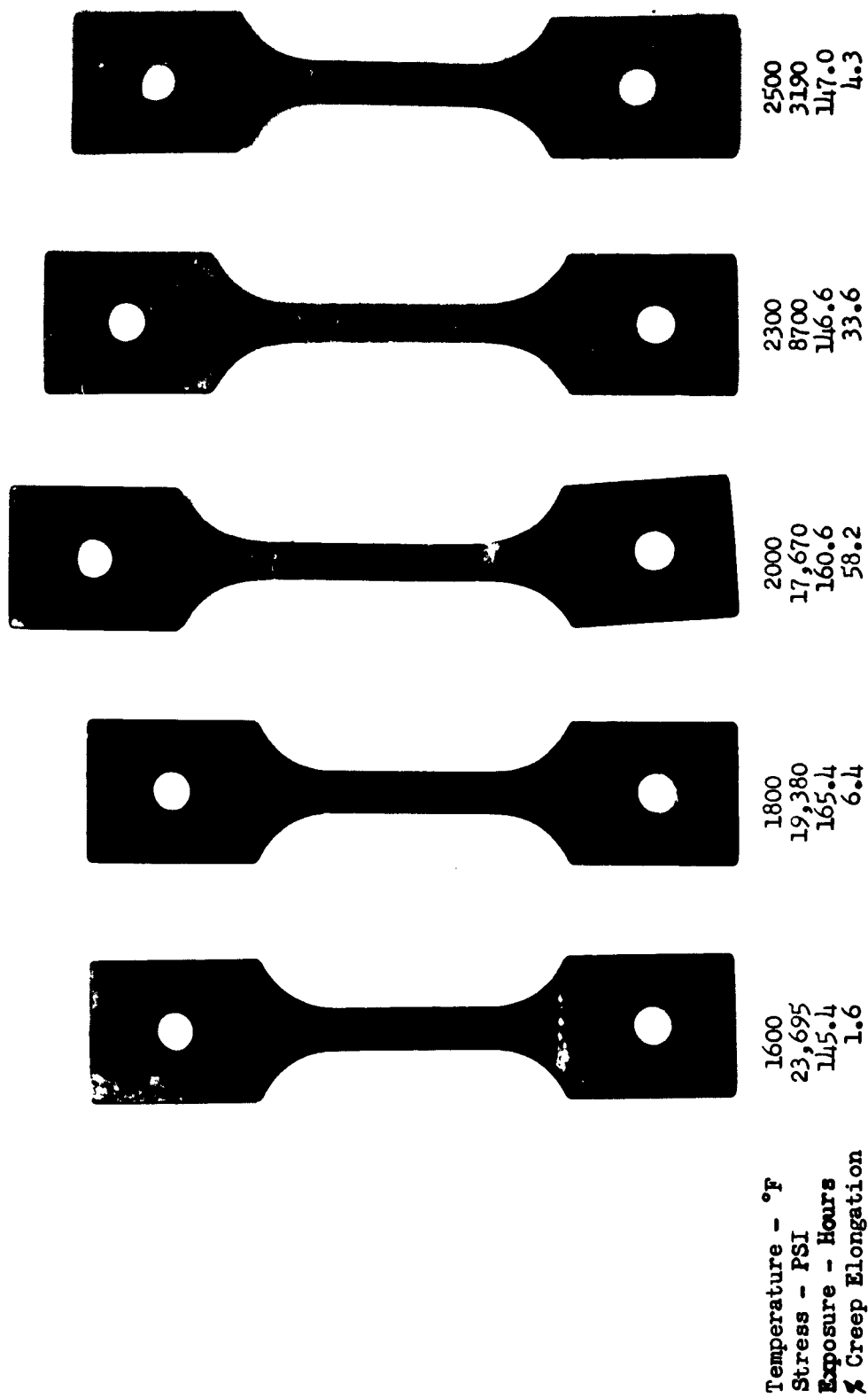


Figure 51 TRW Coated FS-85 Alloy Stress Rupture Specimens Stress Oxidation Tested Without Failure in Air at 1600-2500°F

the stress oxidation exposures on the mechanical properties of the coating-base metal systems. These data are tabulated in Tables 18 and 19. In general there were no significant alterations in the strength or ductility of the coated FS-85 alloy at room temperature as a result of the stress oxidation exposures in air at 1600-2500°F up to 150 hours in the absence of coating failure. The slight increase in strength or reduction in ductility of a few specimens is undoubtedly associated with a limited amount of oxygen penetration through the coating. All of the mechanical property calculations were based on the gage section dimensions measured after creep.

#### 5.8 Conclusions - Design Data Study

- 1) Cyclic oxidation tests of Pfaudler and TRW coated FS-85 alloy in air at 1600-2600°F indicated that the TRW coating was more protective on FS-85 alloy at temperatures above 2000°F.
- 2) Rapid thermal cycling of Pfaudler and TRW coated FS-85 alloy for 100 to 500 cycles from 2600 to 250°F by oxyacetylene torch heating and air blast cooling was detrimental to the protective properties of these coating-base metal systems upon subsequent exposure in air at 1600°F.
- 3) The tensile properties of uncoated FS-85 alloy sheet tested in vacuum at room temperature to 2600°F indicated the presence of a temperature dependent strengthening mechanism in the range 1200-1600°F. A corresponding minimum in the ductility of uncoated FS-85 alloy in the 1200-1600°F range was associated with this strengthening effect.
- 4) The combination of coating application thermal treatments and the presence of brittle coating surface layers virtually eliminated the strengthening effect observed in the 1200-1600°F range for the uncoated FS-85 alloy sheet. At all other test temperatures from room temperature to 2600°F, application of the Pfaudler and TRW coatings to FS-85 alloy had no significant influence on the alloy strength.
- 5) The presence of the brittle coating layers drastically reduced the measured ductility of FS-85 alloy on tensile testing in the 1200-1600°F range, indicating severe notch sensitivity of the alloy in this temperature range.



TABLE 18  
Room Temperature Tensile Properties of Stress  
Oxidized Pfadler Coated FS-85 Alloy Stress Rupture Test Specimens

Temperature °F	Stress Rupture Test Conditions			Tensile Properties			
	Stress PSI	Hours Exposed	Creep = % Elongation	UTS PSI (1)	0.2% Offset Y.S. = PSI	% Elongation (2)	% R.A. (3)
As Coated							
1600	13540	164.2	0.25	89500	70100	16.7	16.6
1600	13540	162.9	0.25	79700	64900	7.4	8.1
1600	20310	163.0	0.0	90200	70500	10.0	9.6
1600	23695	159.7	0.2	86800	64300	18.2	27.2
				86200	68700	7.2	0
1800	12920	162.5	1.3	82150	64100	11.5	31.1
1800	19380	162.8	3.7	83350	63550	18.4	42.6
1800	19380	163.0	2.3	82900	62300	20.0	41.7
1800	22610	145.4	9.9	83700	64150	15.0	48.5
1800	22610	159.7	10.3	83100	66300	16.5	50.0
2000	11780	146.8	2.5	80350	61500	20.5	52.7
2000	11780	146.0	17.7	86050	68150	17.8	51.9

(1) Strength calculations based on approximate load bearing cross section determined from gage length extension at constant volume.

(2) Percent elongation based on creep elongated initial gage length.

(3) Reduction in area based on creep reduced initial cross sectional area.

TABLE 19  
Room Temperature Tensile Properties of Stress Oxidized TRW Coated  
FS-85 Alloy Stress Rupture Test Specimens

Stress Rupture Test Conditions				Tensile Properties			
Temperature °F	Stress PSI	Hours Exposed	Creep - % Elongation	UTS PSI (1)	0.2% Offset		R.A. (3)
					Y.S. - PSI	Elongation (2)	
As Coated				79300	63700	27.5	64.5
1600	13540	163.2	0.6	81600	65200	7.0	54.8
1600	20310	162.4	1.1	81450	63200	20.6	53.8
1600	23695	145.4	1.6	83000	64400	20.9	62.6
1800	12920	164.3	1.6	81950	63500	17.2	44.9
1800	19380	145.4	5.7	83300	61450	17.5	40.3
1800	19380	165.4	6.4	84800	64900	17.2	50.7
2000	11780	145.1	3.2	80950	62200	19.2	59.5
2000	17670	160.6	58.2	91800	79850	3.0	8.0
2300	8700	145.8	30.5	90650	72450	10.2	46.5
2300	8700	146.6	33.6	80850	66350	16.1	46.8
2500	3190	147.0	4.3	75200	61400	20.3	55.4
2500	3190	159.0	5.3	74350	51500	18.9	62.6

(1) Strength calculations based on approximate load bearing cross section determined from gage length extension at constant volume.

(2) Percent elongation based on creep elongated initial gage length.

(3) Reduction in area based on creep reduced initial cross sectional area.



- 6) The generally lower ductility of Pfudler and TRW coated FS-85 alloy sheet tested in air as compared to the uncoated sheet tested in vacuum is also attributed to crack propagation from the brittle surface layers; however, the overall effect is less drastic in regions where the base metal exhibits less notch sensitivity.
- 7) Both Pfudler and TRW coating - FS-85 alloy systems tested in air exhibited comparable tensile properties in the room temperature to 2600°F temperature range.
- 8) The Pfudler and TRW coating applied to FS-85 alloy will tolerate tensile prestrain into the substrate plastic deformation range at all temperatures from room temperature to 2600°F without the loss of 2 hour oxidation protection at 1600 and 2600°F.
- 9) At 2000°F and above both coatings were sufficiently plastic to tolerate tensile prestrain without loss of protective properties to strain levels far in excess of practical design limits.
- 10) The stress rupture properties of Pfudler and TRW coated FS-85 alloy were nearly comparable at temperatures from 1600 to 2300°F, whereas, the TRW coating provided greater stress-oxidation protection at 2500 and 2600°F.
- 11) Comparison of the non-thermal cycle stress rupture lives with the cyclic oxidation properties of the Pfudler and TRW coating on FS-85 alloy indicated that thermal cycling reduced the ultimate protective lives of the Pfudler and TRW coatings.
- 12) At temperatures of 1800°F and above the creep deformation required to initiate coating failure or to produce creep rupture of FS-85 alloy was far in excess of that practical in design considerations. This suggests the more practical significance of creep rate determinations for coated columbium alloys as opposed to determining stress to rupture properties.
- 13) The stress rupture specimen and tensile specimen for the determination of prestrain tolerance must be designed such that virtually all tensile deformation is confined to the specimen gage section.
- 14) Coating defects play a significant role in the determination of all design properties of coated columbium alloys, and must be considered in evaluating the reliability of the overall design characteristics.

6. DISTRIBUTION

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Attn: Mr. David Josephs and Mr. Eli Bradley
29. Applied Physics Laboratory, 8621 Georgia Avenue, Silver Spring,  
Maryland, Attn: Mr. Maynard Hill
30. The Marquardt Corporation, 16555 Saticoy Street, Van Nuys,  
California, Attn: Mr. M.J. Albom